

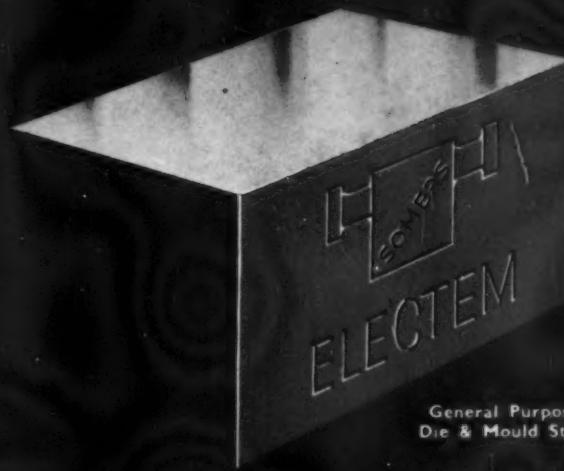
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Vol. 27 : No. 179

AUGUST, 1960

Price 2/6

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Hot Working & Forging
Die Steel



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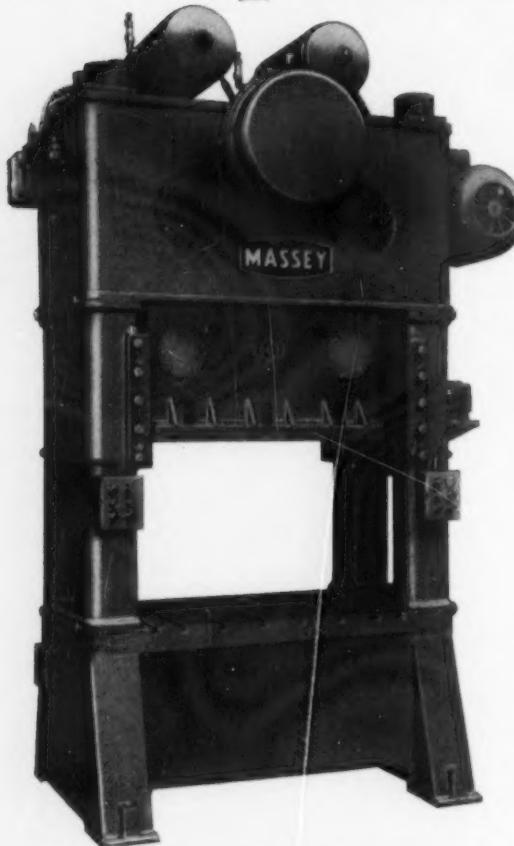
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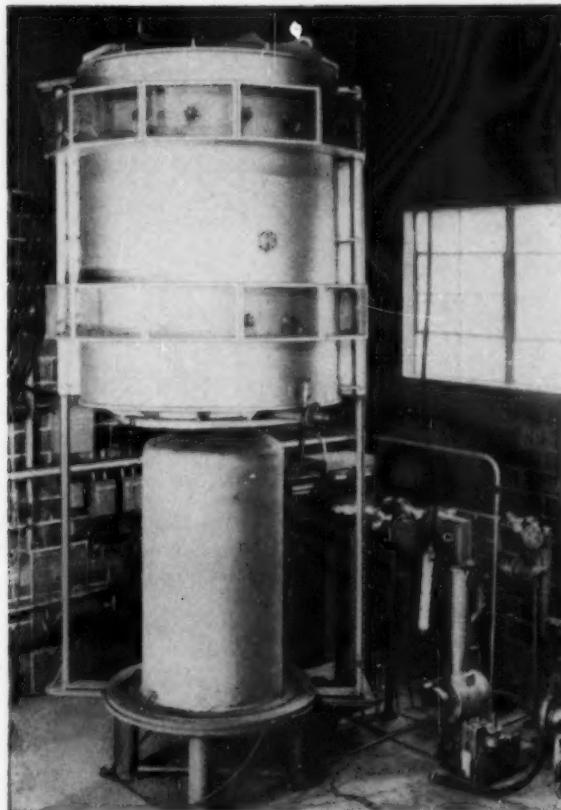
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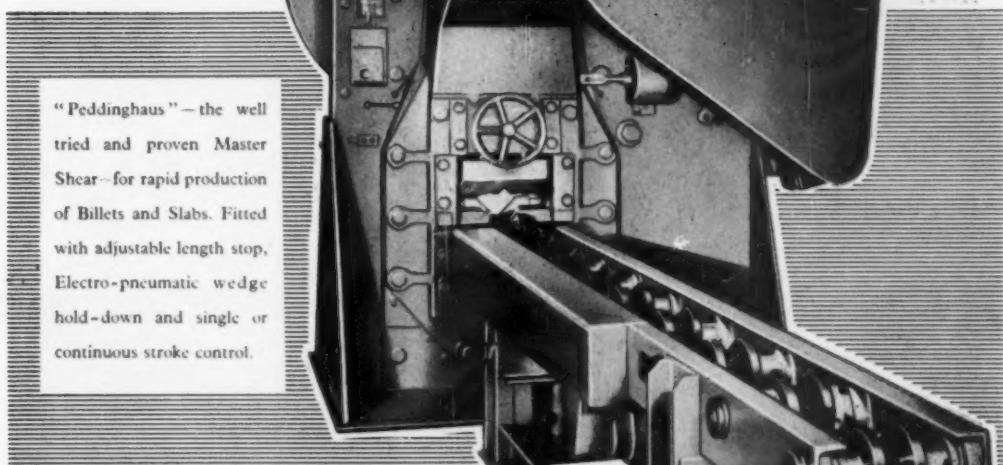


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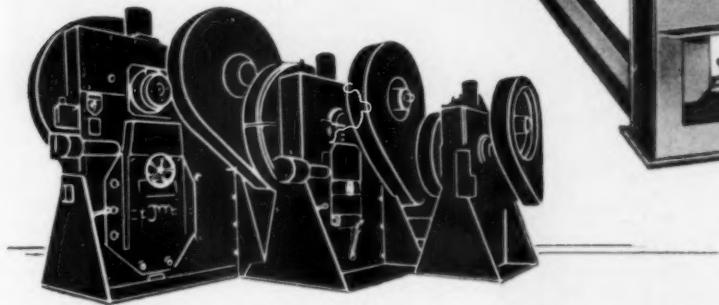
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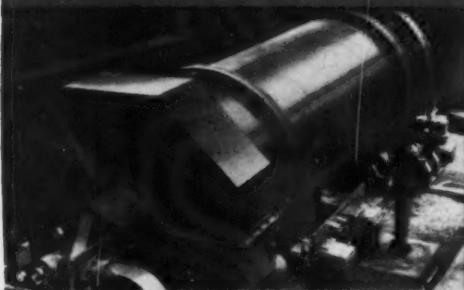


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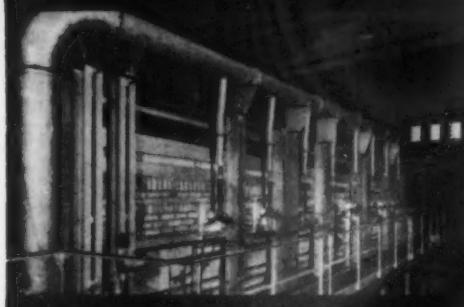


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7

metal treatment
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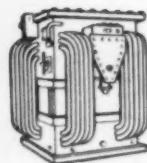
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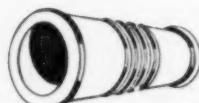
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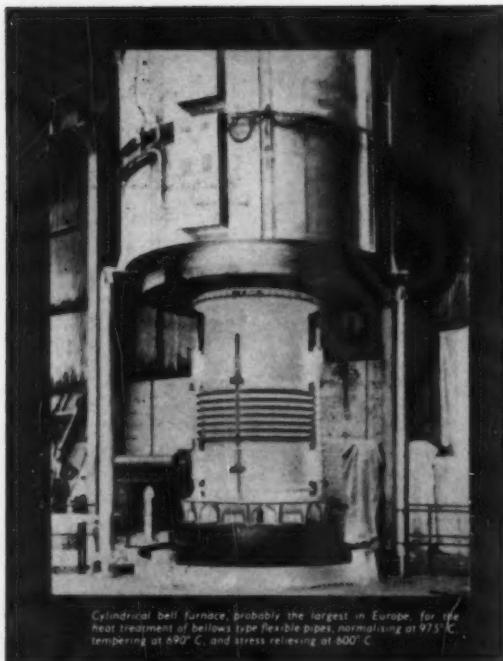
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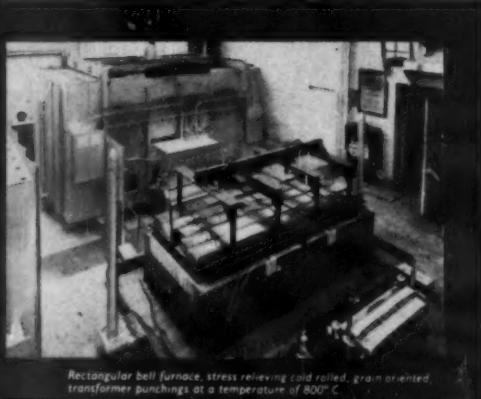
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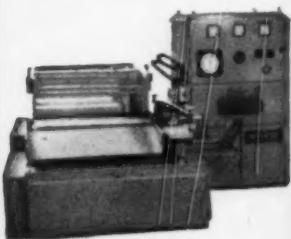
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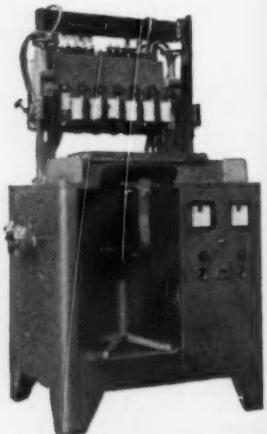
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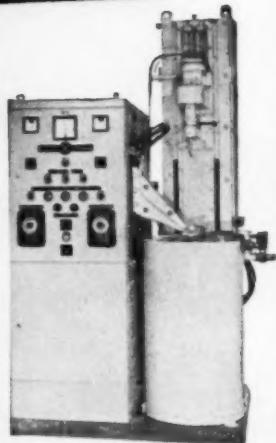


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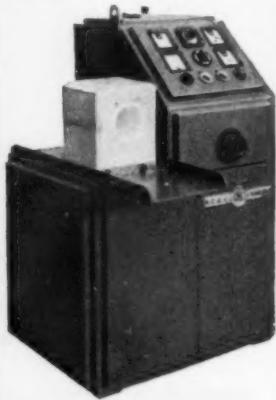
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For heating of billets, pins, tubes and bars of all shapes and sizes,

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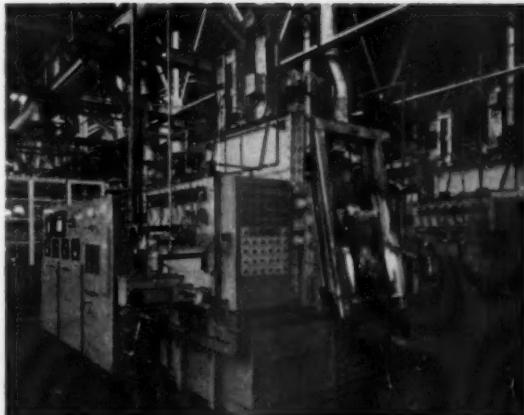
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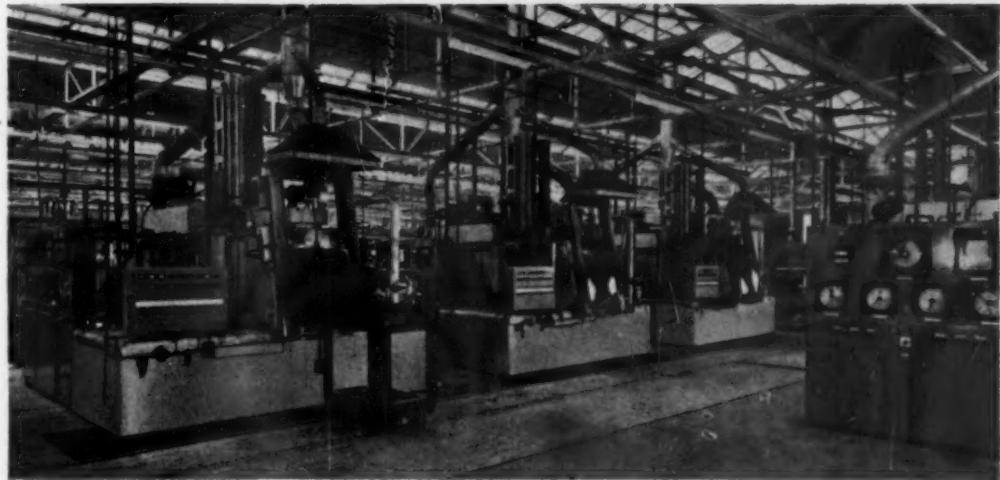
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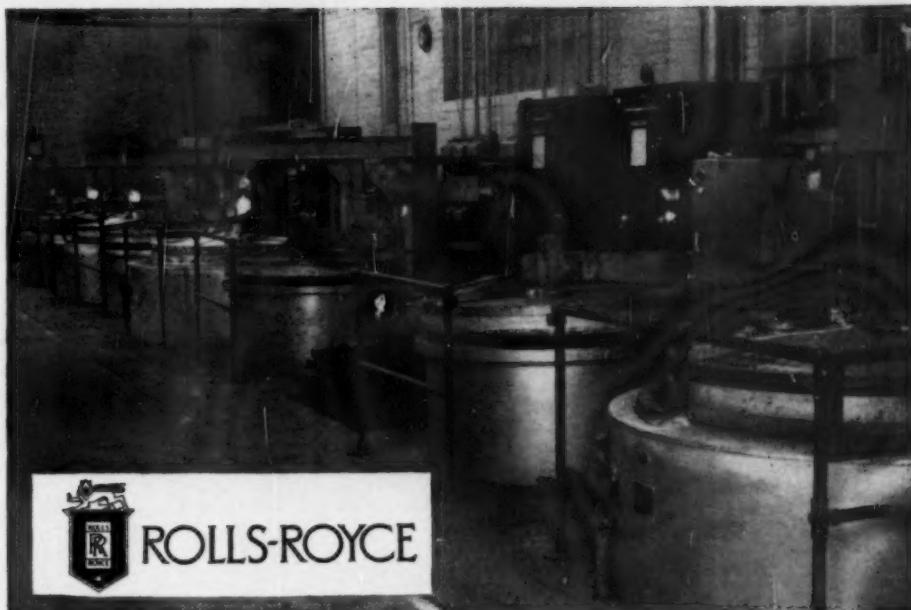
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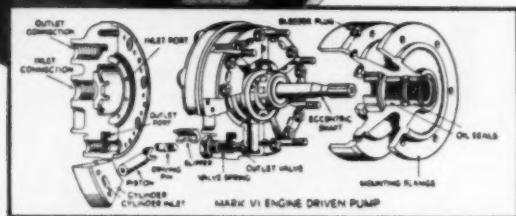
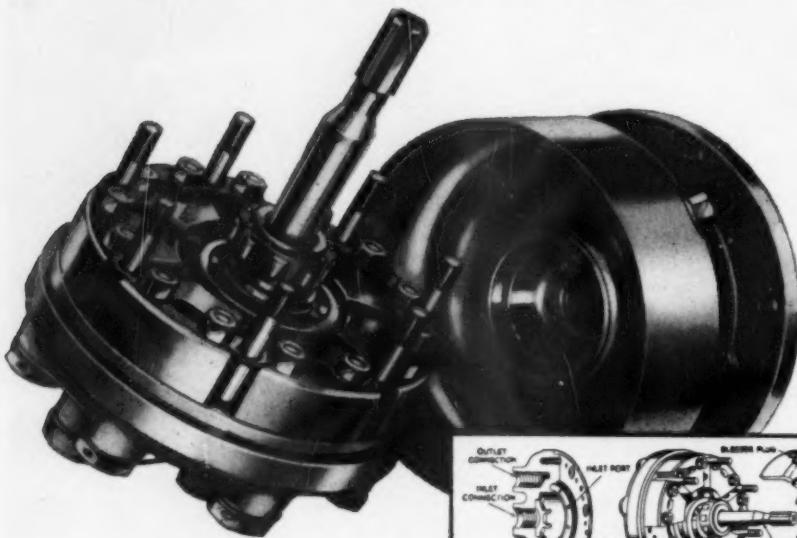
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1 1/2" dia.	Oil quenched 860 C. Water quenched 770 C.	56.3	21.5	83
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The benefits to be gained from the more highly alloyed case-hardening nickel steels, such as EN 33; EN 34; EN 36 and EN 39 include ease of heat-treatment, minimisation of processing distortion, and general reliability.

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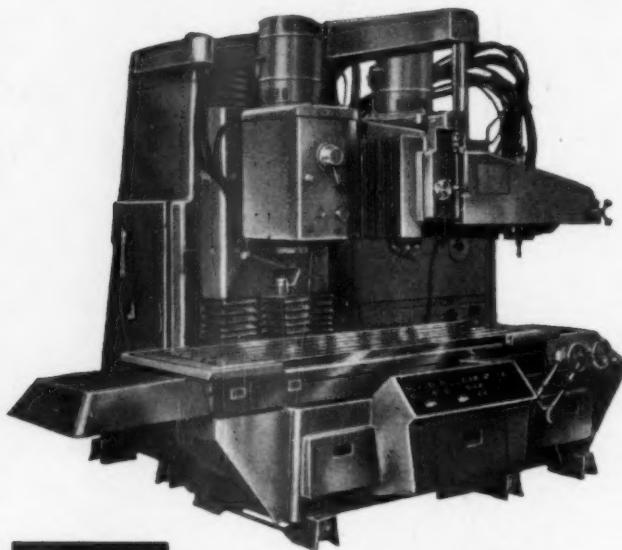
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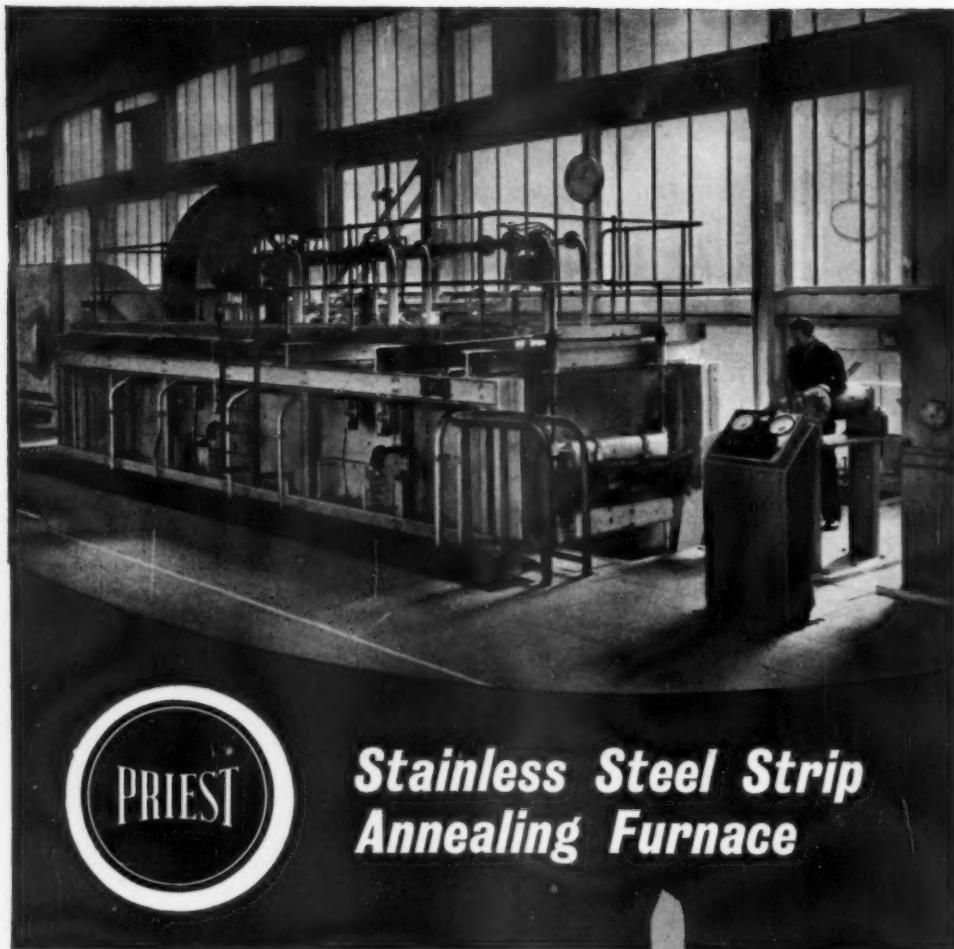
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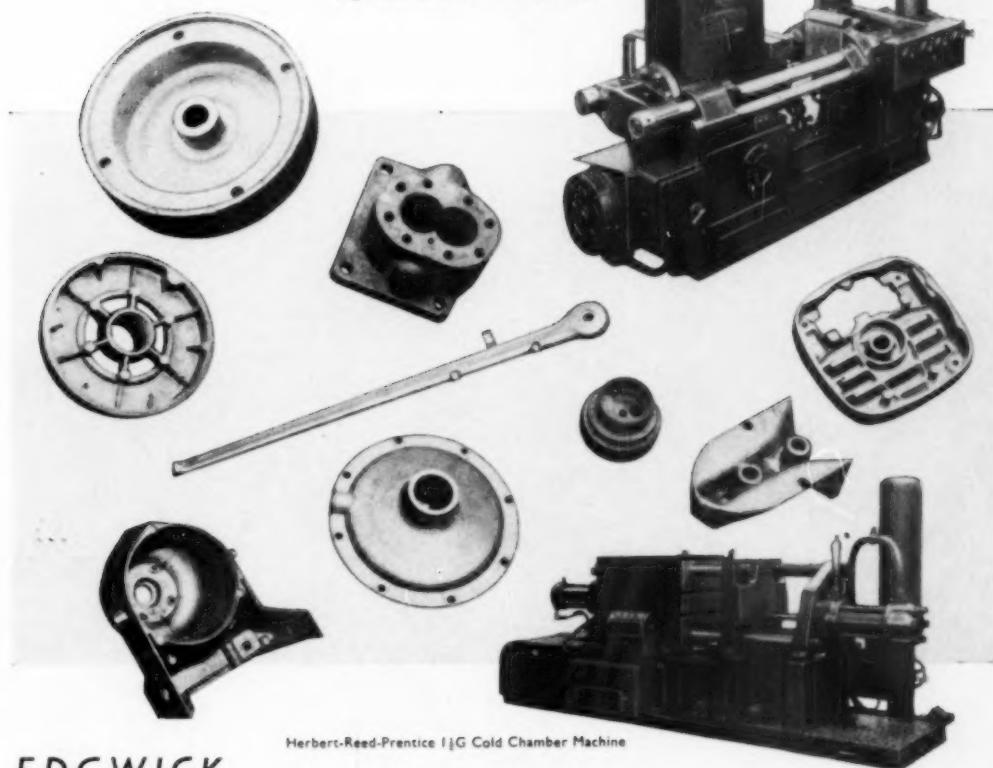
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Casting Capacity, lb.	50	250

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	*1½Z 327	1½G 79	2G 179
Locking Pressure, tons	250	250	400

*Hot chamber type

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Casting Capacity, oz.	6	12

We can offer auxiliary equipment including bale-out furnaces

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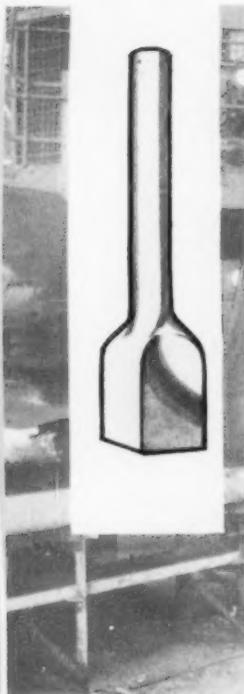
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Left: Photograph showing 29 ton ingot being forged into saw blocks from E.N. 16A steel.

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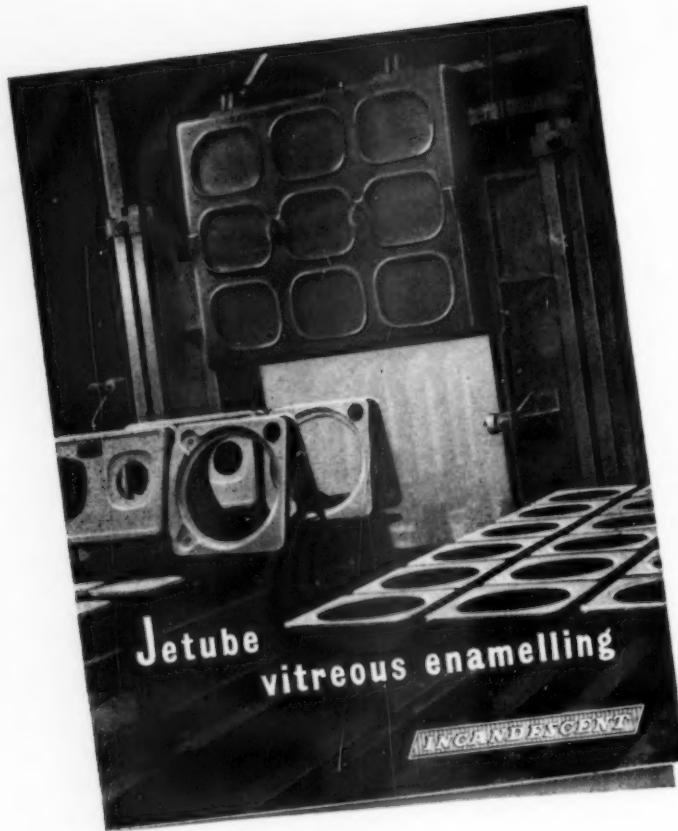
Right: Saw block being quenched.

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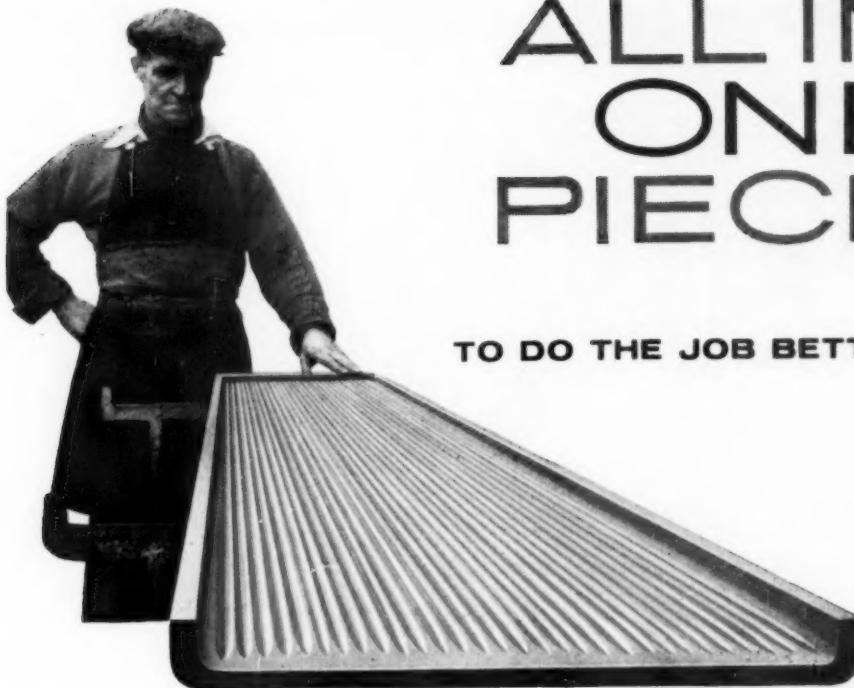
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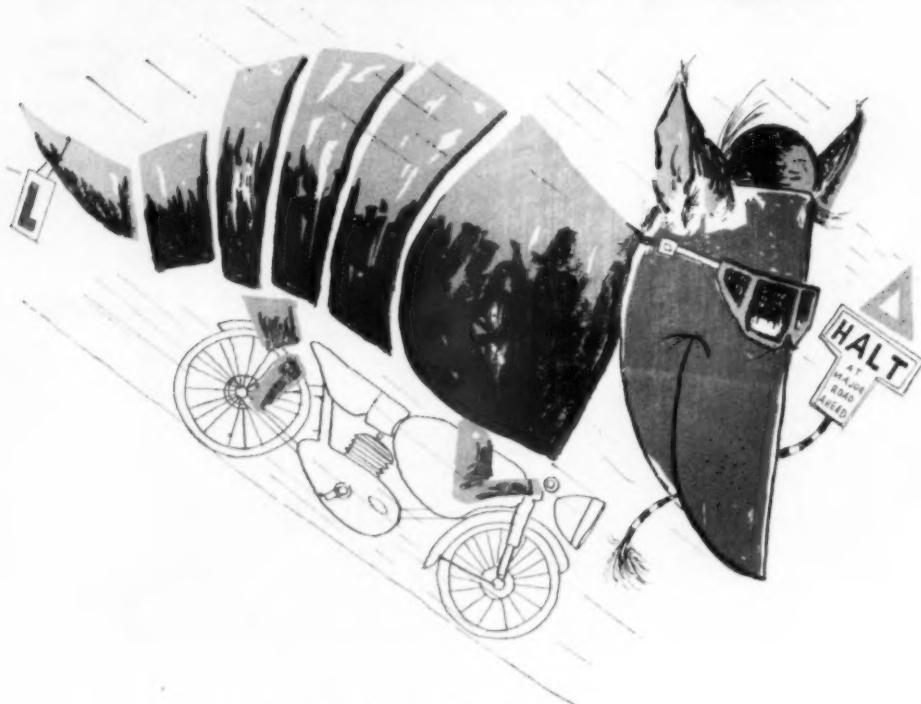
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August 1960
Vol 27, No 179

Metal treatment

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This journal is devoted to metals—ferrous and non-ferrous—their manufacture, properties, heat treatment, manipulation, testing and protection, with research work and development in all these fields

305 **Status of the metallurgist**

306 **Experimental forge furnace**

307 **Metallurgy in nuclear power technology** 2. Casting and working uranium and thorium J. C. WRIGHT, B.Sc., Ph.D., A.I.M. The metallurgy of nuclear power materials is developing on such a wide front and so rapidly that it is difficult for the non-specialist metallurgist to keep abreast with its scope. Dr. Wright outlines the subject in a series of articles

317 **Prespheroidizing**

319 **Application of electron microscopy** Investigation into the subgrain structure of high-purity aluminium DR. H. BICHSEL The subgrain structure of roll-hardened and annealed high-purity aluminium foil has been investigated by transmission electron microscopy. It is shown that a cell structure arising after polishing comes from local etch pits and is not directly connected with the subgrain structure

326 **Books**

327 **The solidification of alloys** A. KOHN AND J. PHILIBERT Minor heterogeneity in alloys is the direct result of the process of solidification. The work done during the last 10 years, notably in the field of the structure of metals in the liquid state, and of phase transformation phenomena, enables more precise explanation to be made

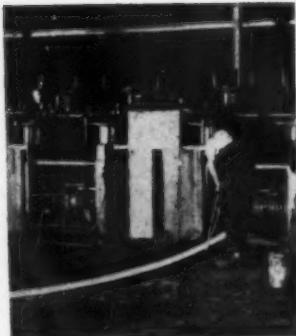
335 **Electrothermal stress relieving**

338 **Fully automatic heat treatment**
Installation at British Timken's Daventry Works

341 **News** 342 **New films**

343 **People** 345 **Instrumentation** 347 **New plant**

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Status of the metallurgist

ONE of the most outstanding changes in modern society has been the emergence of the technologist as an individual of responsibility. There have been other ages and societies when the fabricator of metals was held in high esteem, and up to quite recent times in one Eastern society, the goldsmith was considered to be the equal of the highest caste. The fact that the technologist suffered an eclipse during recent European history should, perhaps, make us look to our laurels in this more favourable period.

In his presidential address to the Institution of Metallurgists last May, Mr. W. E. Bardgett, research manager, The United Steel Companies Ltd., considered the importance of training, equipment and buildings in the standing of the metallurgist. On the subject of training he suggested that senior metallurgists should have an important voice in interpreting the needs of the apprentice or trainee. Metallurgists had a responsibility to their juniors in providing every possible facility for expanding their activities and experience in a planned arrangement. The first two years of the school leaver should, as far as possible, be spent on a very general apprenticeship, moving from one department to another, to gain an idea of the scope of each branch of technology before finally deciding to adopt metallurgy as a career.

It had to be accepted that organized training was as important for the graduate as for the junior metallurgist. One should aim for an 'apprenticeship' period of about two years, at the end of which the graduate would decide whether his interests lie in research or in works metallurgy. The major portion of the training should be in the research laboratory under the supervision of senior metallurgists doing metallurgical work. He should, during his training, also have an opportunity to study the detailed activities of the works metallurgists in relation to works processes.

Apart from the question of providing larger numbers of trained and qualified metallurgists to meet the present and future normal demands, Mr. Bardgett said that his general experience was that there was often a tendency to limit metallurgical effort in works laboratories to an undesirable low level. We had faith in the value of the contribution which metallurgical science could make and a little empire building might, in certain cases, be justified.

Referring to equipment, Mr. Bardgett said that the tools of the metallurgist were important from many aspects: it was on them that our contribution to training, quality of work, and in fact our standing as practical metallurgists largely depended. It was not always easy to justify expenditure on equipment, particularly when commercial considerations call for assessment of return. He instanced the example of the electron microscope and said that it had been with us for a long time and some people might be put off its use through the limited amount of useful information which had resulted, but the relatively recent advance in instruments and techniques had completely changed the picture. Our policy in the matter of equipment should be a bold one. First-class equipment, continued Mr. Bardgett, was incompatible with poor surroundings, and whilst it would seem obvious to say that our laboratories, dealing with fine work, should be clean, healthy and comfortable, experience indicated that the comment was not out of place. Maintaining or raising the standards of our environment was an important objective. To have a laboratory of good design and well equipped, attracted not only more people to the profession but also a better type of person.

Mr. Bardgett believed the profession could be enriched by a deeper appreciation of the importance of training, equipment and buildings. Whilst high standards might be available to some, a consciousness of maintaining them should have an important place in our minds. To those who were not so well placed, constant persistent endeavour to effect improvement should always be pursued.

Experimental forge furnace

New installation at BISRA

A FULLY-INSTRUMENTED experimental forge furnace has just been completed in the Energy Section of the British Iron and Steel Research Association, Battersea Laboratories, London.

The furnace will be used to investigate the thermal efficiencies attainable in batch-type heating processes. It is designed so that the material of construction can be varied, as can the heating schedule, type of fuel and furnace load, recirculation and waste heat recovery techniques.

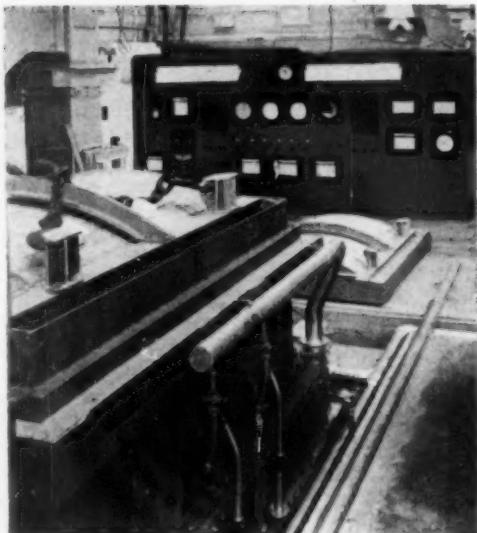
The heat capacity of the chamber can be altered by the provision of two alternative roofs, one of fire-brick and the other of high-temperature insulating brick. Oil or gas firing is provided for by means of three burners firing tangentially above the ingot and two below. The effects of convection and radiant heating can be studied.

Automatic temperature and flow control

Comprehensive instrumentation allows calculation of complete heat balances at short intervals throughout the heating cycles. The furnace temperature is measured by means of platinum/platinum-rhodium thermocouples, and is indicated and recorded on a Honeywell ElectroniK recorder-controller. Integral in the instrument is a pneumatic control unit which proportions a control signal between 3 and 15 lb. sq. in. The output from this primary controller is applied to the set points of the gas, air and oil flow controllers. The oil flow is measured by means of a transmitting Rotameter and the flow controlled by a diaphragm valve. Gas and air flows are measured by orifice plates and the pressure drop transmitted by a differential converter to the flow controllers, which pneumatically operate butterfly valves in the pipelines.

The primary temperature controller is programmed to give the required heating schedules, the desired preset fuel-air ratio being automatically maintained by the secondary flow controllers.

Over 80 thermocouples are embedded in the furnace walls and 12 within the 3-ton ingot. These, together with further thermocouples in the furnace chamber, flues, air ducts, recuperator and stack, will be used to calculate the distribution of heat during operation. All these thermocouple readings are continuously recorded on four Honeywell 12-



Experimental forge furnace and instrumentation panel at BISRA, Battersea Laboratories

point strip chart recorders, in conjunction with switchgear. A digitizing system is applied to two of the temperature recorders so that the bulk of the information is recorded on punched tape to facilitate direct calculation using a Pegasus computer.

Pressure and waste gases

In order to minimize the leakage of cold air into the furnace chamber a pneumatic butterfly valve is fitted in the stack and the pressure held close to a set point by means of a Honeywell pressure controller and the pressure is recorded continuously on an Evershed and Vignoles electric pressure recorder.

The flue gases are continuously analysed. A Cambridge katharometer with a Honeywell strip chart recorder and an Infra Red Development Co. infra-red analyser are used to estimate carbon dioxide, whilst a Kent paramagnetic analyser measures the oxygen content of the flue gases.

Scientific equipment exhibition

The Griffin and George Group of Companies, Ealing Road, Alperton, Wembley, Middlesex, held in July what is claimed to be one of the largest exhibitions of scientific equipment ever organized in the United Kingdom by one firm.

The equipment exhibited comprised not only the latest equipment developed by Griffin and George, but also that of over twenty manufacturers whose equipment is sold by Griffin and George (Sales) Ltd., in many instances throughout the world. Also, much interesting equipment from overseas was shown.

Metallurgy in nuclear power technology

2. Casting and working uranium and thorium

J. C. WRIGHT, B.Sc., Ph.D., A.I.M.

The metallurgy of nuclear power materials is developing on such a wide front and so rapidly that it is difficult for the non-specialist metallurgist to keep abreast with its scope. Dr. Wright, Reader in Industrial Metallurgy, College of Advanced Technology, Birmingham, outlines the subject in a series of articles which are appearing monthly in this journal

CASTING URANIUM METAL. Remelting of the uranium biscuit resulting from the reduction process is necessary to eliminate impurities such as slag particles and hydrogen. The solid impurities tend to float out of the liquid metal and, since the melting is generally carried out under vacuum, gaseous impurities are greatly reduced. To consolidate metal which has been extracted from the hydride and electrolytic processes, the powder or crystals are first compacted and then melted.

In a few special cases, the uranium billet produced in the final extraction vessel is satisfactory for direct processing, but fuel elements made this way contain hydrogen picked up during the extraction process. A better result is obtained by remelting and vacuum casting. Most uranium cast in this way is melted in a high frequency electric, bottom-pouring type of crucible furnace. The heat is generated normally in a graphite crucible by means of an induction coil outside the vacuum vessel. At the casting temperature of 1,200-1,300°C. (uranium melts at 1,130°C.), a stopper in the bottom of the crucible is pushed up so that it floats on the melt. The metal flows through the tap hole, usually 300-500 lb./charge, via a launder to a billet mould below (fig. 3). The whole assembly is maintained under vacuum; high vacuum for special cases, but for most purposes a pressure of 0.5 mm. Hg is satisfactory. The vacuum process removes considerable metallic and non-metallic inclusions. Any oxide and fluoride present floats to the top and the hydrogen content is reduced to about 2 p.p.m.

The process can be applied to casting simple shapes, including fuel rods, direct, without going through a mechanical working procedure. It is possible to confine the solidification pipe to the top 3 in. of a 29 in. by 1½ in. diameter bar. Inclusions

are minimized by the addition of a small amount of magnesium to the initial charge.

Vacuum melting and casting in a molybdenum-wound resistance-heated furnace has been achieved, using a tilting furnace and steel or graphite moulds.

Typical analyses of variously produced uranium are given in Table 3. The purities are adequate and no materials with high thermal neutron absorption properties are present.

Heavy uranium ingots weighing 1,200 lb. can be made by melting in an open H.-F. induction furnace. The metal is melted under an argon atmosphere and covered with a $\text{BaCl}_2/\text{CaCl}_2$ flux as it melts and during pouring. Graphite or MgO crucibles and graphite tundishes are used and a recovery of 91-95% is claimed.*

H.-F. melting with a graphite crucible, which acts as a susceptor as well as a container, and lip-pouring into a mould, the whole assembly being in a vacuum or inert atmosphere tank, has also been used.

Mould materials and crucible materials. Uranium is usually melted in graphite crucibles suitably coated to avoid carbon pick-up. The most popular coatings are magnesia and alumina. Common electrode graphite is easily machined to any required crucible shape. It does react with uranium to form a carbide, but the reaction is slow below 1,600°C., and the amount of carbon likely to be introduced at lower temperatures is not likely to be serious in commercial quality metal. Lime crucibles are in use for melting super-purity uranium. Uranium oxide, beryllia, zirconia and thoria are also non-

*G. W. P. Rengstorff and H. W. Lowire, *Metal Progress*, 1957, 72 (3), 76.

reactive but they are all expensive and very brittle which makes them commercially unattractive.

For mass production of simple shapes the most satisfactory mould material is mild steel protected by a refractory dressing, preferably alumina, which also acts as a thermal barrier during solidification and prevents cold shuts. Very high-quality casting surfaces and close limits of accuracy may be obtained. Graphite is an alternative mould material.

Casting and ingot quality. The high sp. gr. of uranium assists feeding of castings and, if top feeding is used, less feeding metal is required than for most metals. Casting of 1 in. dia. bars can be arranged so that metal flows into the mould at a rate which keeps pace with the solidification so that feeding takes place continuously during freezing. Uranium has a low latent heat of fusion and it solidifies rapidly and there is little chance for oxidation once it is in the mould when using non-controlled atmospheres.

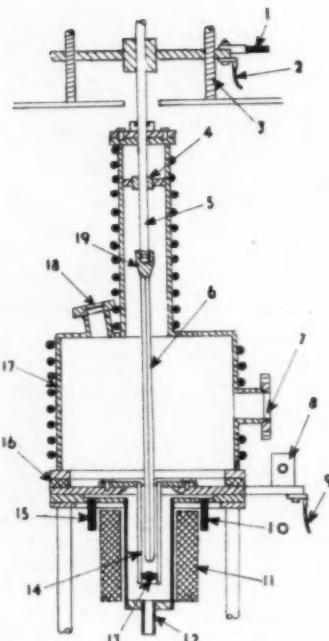
Simultaneous arc melting/casting. This is a possible competitor to the vacuum high frequency electric furnace. Two types are possible: (1) Non-consumable electrode (tungsten) in an argon atmosphere or vacuum electric arc furnace; and (2) consumable electrode of uranium in an argon atmosphere or vacuum electric arc furnace (fig. 8).

The former is not suitable for large ingots because the degree of control over the arc is insufficient to ensure freedom from tungsten contamination but it may be used for laboratory heats.

The consumable electrode method uses either cast or worked bar or assembled powder compacts as the electrode. Both methods produce simple shapes by feeding from the arc direct to a water-cooled copper mould. The limitations are: (1) Only simple shapes can be cast; (2) a large machining allowance may be needed; (3) it is necessary to start with an electrode made from an alloy or mixture of the composition required; (4) since the mass of metal molten at any time is small, there is little opportunity for removal of volatile solids or floatable impurities; and (5) remelting is usually necessary to improve the homogeneity of the alloy.

Fabrication (general)

Fabrication may be carried out on cast or powder compacted uranium. The problems concerned are



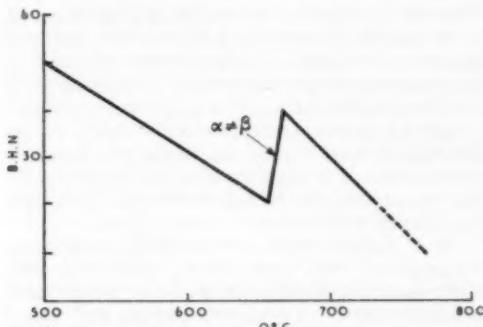
8 Schematic diagram of a consumable-electrode arc-melting furnace (USAEC Report BM1/1142)

1, - terminal to rectifiers. 2, - to electrode controller. 3, Electrode drive. 4, Electrode guide. 5, Water-cooled copper electrode. 6, Consumable electrode. 7, Vacuum flange. 8, - to rectifiers. 9, + to electrode controller. 10, Water outlet. 11, Electromagnetic coil. 12, Water inlet. 13, Starter charge. 14, Copper crucible. 15, Water outlet. 16, Insulating ring. 17, Furnace shell. 18, Sight glass. 19, Threaded adapter.

dominated by the high reactivity of uranium and its alloys. They are oxidized easily and also attack container and die materials readily. Much fabrication requires a high temperature and, to avoid atmospheric reaction, either a vacuum or protective atmosphere is required. This is particularly so with powder metallurgy methods which are attractive in the handling of reactor fuel materials; but their extreme reactivity, together with the large surface areas of the powders, requires the processes to be meticulously controlled. Press capacity limits the size of fuel element which can be made readily by conventional powder techniques, and this has led

TABLE 3 Analyses of uranium metal (in p.p.m.)

Uranium	C	N	O	Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Si
Mg-reduced . . .	8-13	19	—	5	20 20-50	5	1	20 50-200	15	1 5-10	10	15
Ca-reduced . . .												50-100
Mg — reduced, remelted, graphite . . .	240-295	10-16	6-16	10	20	2	5	30	0.5	2	15	15
Ca — reduced, remelted, alumina . . .	40	7	16	20	—	5	3	28	5	8	5	10



9 Variation in hardness of uranium with temperature

to work on hydrostatic pressing which is being developed to include hot pressing.

Uranium in ingot form may be fabricated into useful shapes by most of the conventional metallurgical forming operations—forging, swaging, rolling, extruding, drawing, etc. These operations can be carried out either cold or hot but preferably below 600°C. Extrusion in massive form requires higher temperatures. During hot working, suitable precautions must be taken to prevent contamination of the uranium and reaction with air at elevated temperatures. This may be accomplished by using salt or oil baths or inert atmospheres during heating for forming, and during subsequent annealing if this operation is included.

Fabrication of uranium shapes may be accomplished by either welding or brazing, although the methods are not entirely satisfactory. Special precautions are required to prevent oxidation of the uranium. Most of the commercial brazing materials alloy with uranium to form brittle intermetallic compounds at the brazed joint. This does not give a metallurgically sound joint and may be prevented to some extent by first electroplating the uranium with silver or nickel.

Shaping uranium by conventional machining methods is relatively easy. Owing to the pyrophoric and toxic characteristics of uranium it is essential that a suitable lubricant be used. If machining is done dry, adequate atmosphere exhaust facilities are necessary.

Deformation characteristics of uranium

Deformation of uranium up to 450°C. takes place mainly by twinning and work hardening occurs. Over this temperature range, a preferred orientation, (010), is developed in the material, along the major axis of working.

At higher temperatures the twinning mechanism is replaced by a slip mechanism and at the same time annealing and recrystallization become pos-

sible. As the working temperature is increased above 450°C. a dual texture develops with both (010) and (110) planes in the direction of working and as the working temperature increases towards 600°C. the (110) direction becomes dominant. Uranium may be hot worked in this range and the metal gets progressively softer as the temperature is raised. The minimum recrystallization temperature for heavily cold worked uranium is about 425°C.

In the β -phase, 660-770°C., the forces required to hot work the metal are two to three times as great as at 650°C., and the ductility is substantially less. Over this range, the worked uranium distorts by a slip mechanism. In the γ -phase above 770°C., the structure of uranium is B.C.C.; it is too soft and plastic to retain its shape properly in many types of forming operation unless supported. However, extrusion may be carried out conveniently and with relatively little power in the γ -state. The variation in the hardness of uranium is shown as a function of temperature in fig. 9.

Phase transformations should be avoided in hot working uranium. If a phase transformation occurs in uranium during deformation, there will be a tendency for it to break up because of the severe stresses created by volume changes during the transformation superimposed on the working stresses.

Preheating for working

Uranium reacts increasingly with air as the temperature rises but many working operations can be carried out without protection from oxidation. This applies to operations such as rolling and die forging which may be carried out rapidly in the high α range and in which the die or roll pressure compacts the initial porous oxide film into a dense protective layer.

Protection during preheating is general, however. The salt bath is the most convenient medium and eutectic lithium and barium carbonates or lithium and potassium carbonates have been found generally suitable.

When a salt bath is inconvenient it is necessary to provide a controlled atmosphere and the most convenient way is to use metal containers for the billets in a conventional furnace, maintaining atmospheric control within the container only (argon, helium, vacuo).

Uranium work hardens readily and has to be inter-stage annealed if the rolling temperature drops in hot rolling and always in cold rolling. Time in salt baths is minimized as much as possible to avoid hydrogen pick-up which would reduce ductility. During hot working, the salt stays molten, adheres to the bar surface and protects it from the air.

Rolling

Rolling is carried out in the α -phase between 400-600°C., because below 400°C., α -U work hardens too rapidly, and above 660°C. the β -phase is too brittle. If, however, material which has been well-rolled previously in the high α range is allowed to rise in temperature until it enters the β -range, rolling may continue. Above 770°C., in the γ -phase, uranium rolls readily but is too soft for bars to be handled readily owing to their high density and low resistance to deformation. Material rolled at high speed in the α -phase tends to heat rapidly during rolling, due to friction, both internal and between rolls and stock, caused by the large force required to accelerate this high density material. If temperature control is not important, this eliminates reheating but if a controlled structure is required it is essential to return to the heating furnace to equalize at a constant temperature.

Conventional steel rolling mills are satisfactory for producing uranium bar in the α -range, round pass type rolls being preferred because of the importance of compacting the oxide film and also because uranium has little tendency to spread. The average reduction is about 15% per pass.

Rolling of sheet or strip can also be done, although the greater ratio of surface area to volume in sheet compared with bar renders temperature control and inhibition of oxidation more difficult. Where thin sheet or strip is required, it is convenient to start by enclosing the uranium in a less readily oxidizable metal (e.g. Ni, using graphite to prevent alloying). After reduction to about 0.05 in., the uranium is removed from the protective metal and further reduced by cold rolling and annealing, or by pack rolling and annealing. Uranium 0.0005 in. thick may be produced in this way.

Forging

This is quite straightforward at temperatures near 600°C. The metal must be protected against oxidation during preheating but can be forged bare. Pressing can be carried out in the γ -phase at temperatures 800-1,000°C. The major problem here is the same as that for extrusion in the γ -phase, that of finding suitable die materials.

Uranium may be heated to forging temperature in a salt bath and water-quenched after forging to prevent further oxidation.

Swaging of uranium is a convenient way of producing small rods down to about 0.05 in. dia. Swaging is best carried out in the α -range around 560°C. and 10-20% reductions per pass are usual. In cold swaging uranium, it may be necessary to anneal after 30-40% reduction.

Extrusion and drawing

Extrusion is usually carried out in the γ -phase at

800-950°C., when the low resistance to deformation is an asset in keeping the loads on dies and containers to a reasonable limit. Vertical extrusion is used when bending of the weak hot section becomes a serious disadvantage of the horizontal process.

Initially, uranium extrusions were carried out by canning the billet and extruding an integrally canned bar. If canning material can be left on the bar this process may be satisfactory but its removal by dissolution is tedious.

Since uranium tends to alloy rapidly at extruding temperatures with iron, cobalt, nickel and their alloys a principal difficulty is the preservation of extrusion dies. Chromium plating and heavy lubrication by graphite has been used. Additionally, sintered or cemented carbide dies, which are very hard, do not alloy with uranium, but the rough handling they receive is very detrimental. The most satisfactory material for extrusion dies so far has been stellite (45-60% Co, 25-30% Cr, 3-14% W, 2½% C). This can be laid down by fusion on a steel base and cut to form a die. In spite of its high cobalt content it is found satisfactory in practice, due probably to the extreme hardness of stellite preventing deformation which may promote alloying.

Heating for extrusion again uses the salt bath technique, a eutectic mixture of lithium and potassium carbonate being employed. The speed of extrusion after soaking is usually sufficiently high to avoid excessive process oxidation.

The metal extruded as γ -uranium has a very heterogeneous and large grain size, and no characteristic orientation.

If reaction between uranium and extrusion dies becomes too intense, the extrusion temperature may have to be lowered and extrusion carried out in the top of the α -range, 300-600°C. This results in a finer grain size than that for γ -extrusion, and large-scale α -extrusion has been carried out.

Tubes and other shapes can be made by extrusion, but die wear and lubrication are serious problems. Tube shells have been made by rotary piercing in the γ -range.

Drawing. Uranium wire and tube can be cold drawn, or warm drawn at about 150°C., but lubrication is important. A mixture of lacquer, molybdenum disulphide and fumed lead-zinc oxide die lubricant has been used by spraying or brushing on and allowing to dry. Tungsten carbide drawing dies are preferred for their abrasion resistance.

Welding and machining

Uranium can be Argonarc welded provided the pieces are free from scale to start with and a nitric acid dip can be used as a final cleaner prior to welding. Since molten uranium is very fluid, adequate support must be provided and weldments

backed up with chill plates. Direct current with the electrode negative is normal welding practice.

Machining. By taking deep rapid cuts with very sharp hard tools, the work hardening of uranium during cutting may be minimized. Also, advantage can be taken of less rapid hardening and lower strengths at temperatures in the range 150-200°C. The chips as they separate from the metal must be completely covered in coolant to prevent combustion. Carbide-tipped tools are used for most machining operations.

The effects of alloying additions on fabrication

The fabrication of uranium-rich alloys differs from that of the pure metal only by the extent to which the alloying element modifies the three allotropic forms of uranium. No elements having wide solid solubility in α -uranium have been found, therefore, in general, the degree of hardening caused by the second phase in α -uranium governs the extent of deformation.

As far as is known, there are no elements which make the fabrication of the β -phase any more feasible than in the pure metal.

There are a number of elements, such as Mo, Zr, Nb, Vd and Ti which have fairly extensive solid solubility in γ -uranium. In such cases, feasibility of working above 770°C. is increased since the uranium is stiffened and is more handleable at high temperatures. In some instances, the β/γ transition temperature is lowered by alloying and it then becomes possible to fabricate in the B.C.C. phase below 770°C. With some alloys (U + 6 to 15% Mo) it is possible to retain the γ -phase in a metastable state at room temperature and cold working of the B.C.C. phase is then possible.

Inspection

Inspection and testing of fuel element stock for power reactors at important stages of manufacture have the following major functions. (1) Elimination of material possessing discontinuities which block heat flow; (2) eliminating paths for escape of fission products; and (3) assurance of proper size and amount of fissile material.

After cladding fuel elements, the cladding must be defect free to prevent leakage of fission products, to restrain distortion of the uranium as much as possible and to eliminate direct fuel to coolant contact.

Eddy current testing may be used for checking thickness and soundness of cladding; scintillation counters for measuring cladding thickness; radiography for inspecting end closures and for locating the core in the cladding; radiation techniques to determine the homogeneity of the core and the amount of fissionable material in the core; ultra-

sonics to test the bonding, cladding thickness, grain size and flaws.

Powder metallurgy of uranium

Production of uranium powder. There are four basic methods of producing uranium powder: (1) Hydrogenation of uranium chips; (2) sodium reduction of uranium tetrachloride in the vapour phase; (3) the reduction of uranium oxides with calcium or magnesium; and (4) the electrolysis of molten uranium salts.

If hydrogen is passed over uranium at 225°C. the trihydride, UH_3 , is formed. This falls from the uranium as 5-10 μ powder. The hydride decomposes in *vacuo* at 300°C., to give a similar-sized uranium powder, but both reactions can be accomplished almost simultaneously by allowing the freshly-formed hydride to fall into eutectic sodium-potassium alloy at a suitable temperature. It is not easy to remove the uranium cleanly from the liquid metal but this is done by pressure filtering and finally cleaning with butyl alcohol. The purity of the resulting uranium powder, apart from the liquid metal contamination, depends on the purity of the original uranium chips and of the hydrogen.

In the second method, uranium tetrachloride and sodium vapours are sublimed from separate vessels in a reaction chamber at 850-880°C. Uranium and sodium chloride precipitate from the reaction. The sodium chloride can be distilled off or leached with ethylene glycol.

The direct reduction of uranium dioxide with calcium or magnesium involves mixing the oxide and the reductant and heating the charge under a protective atmosphere to about 980°C. The mass reacts and uranium powder is separated from the reaction cake.

The thermoelectrolytic extraction of uranium normally results in a free crystalline form of uranium.

Particle sizes and shapes. Calcium-reduced UO_2 yields uranium powder mainly of 10-17 μ size with about 30% below 10 μ . Magnesium reduction yields mainly 5-10 μ powder. Hydride uranium is mainly about 5 μ and up to 10 μ , but tends to agglomerate. The pyrophoricity of uranium powder depends largely on its surface area to volume ratio. Spherical powders are much less pyrophoric than fluffy powders. Generally, the latter make the most easily handled green compacts and spherical powders tend to make the densest sintered compacts.

Compacting. For high green strength, particles must interlock during compacting. Interlocking powders are either dendritic, such as those resulting from thermoelectrolysis, or porous, consisting of chain-like agglomerates. For cold compacting spherical powders, green strength is sometimes boosted with a binder such as paraffin wax, but

some binders are difficult to remove without contaminating the compact. Cold pressing of uranium spherical powders produces a handleable green compact without a binder. The particles are heavily deformed and any oxide skin is usually sufficiently broken for some cold welding to take place.

When cold pressing is the method of producing a compact for subsequent sintering, a pressure of 60-65 ton/sq. in. may be used.

Sintering. The most economical method of producing uranium compacts, provided a density of not more than 18.5 will suffice, is by sintering cold pressed powder at 1,110-1,120°C. for 4 h. A higher temperature is too close to the melting point of uranium (1,130°C.) for safety and longer times result in a markedly coarsened grain size. The average grain size is 200 μ and the grains are completely randomly oriented.

Another method involves a light pressing operation in the green stage, about 30 ton/sq. in., a moderate sinter giving a density of up to 17.5; and re-pressing the sintered compact cold at 175 ton/sq. in. This operation is followed by an anneal which, since the re-pressing imposes considerable cold working, results in recrystallization. Some voids are also diminished during annealing. The final compact has a density of about 18.5.

Hot pressing and hot compacting. Hot pressing of green compacts and hot compacting of the original uranium powder both yield densities approaching the theoretical (19.1). Hot pressing will produce small grained randomly oriented uranium. If a wax binder is incorporated in the green compact, care must be taken to allow the binder vapours to escape or voids will occur giving low densities. At the same time the wax vapour forms a protective atmosphere. A two-stage operation may be used for this. For example, the wax may be vaporized while the compact is under 4 ton/sq. in. at 350°C. This is followed by 10 ton/sq. in. at 750°C.

Good vacuum during the final stages is necessary for hot compacting of powder and it is necessary for the powder to be free from oxide and slag.

The rate of densification falls off as the compact is heated from the alpha into the beta region because β -uranium is less plastic than α -uranium.

It is possible to achieve densification of uranium powder in all phases, without prior consolidation, by direct extrusion of the powder.

Alloying by powder metallurgy. Powder-metallurgy methods of making uranium alloys are associated with such high recoveries that they are very important for making alloys of enriched uranium, even when conventional melting and casting methods are feasible. Uranium alloys are made either by mixing the elemental powders and hot compacting or sintering, or by direct reduction of the oxides of the constituent metals with calcium.

Bonding of uranium powder compacts. Powdered uranium compacts may be strongly bonded to zirconium sheet by hot pressing at 360°C. for 10 h. Compacts may be bonded to aluminium sheet by pressing for 1 min. if the compact is thoroughly heated to 550°C. Not all metals or alloy mixtures can be compacted around massive uranium because if the compacting temperature exceeds 800°C. grain growth of uranium is excessive.

THORIUM

Thorium is important in reactor technology because it might be used as a 'fertile' material for the production of nuclear reactor fuel. The thorium 232 isotope on neutron irradiation can be converted to uranium 233 which is fissile and consequently far more valuable than the original thorium.

Sources of thorium

Thorium minerals are very stable and tend to be released from igneous rocks by erosion and weathering, being transported and redeposited substantially unchanged. Monazite sands are placer deposits formed in this way and are commercial sources of thorium. The best deposits of monazite are in Brazil and India and there are sizeable deposits in the Dutch East Indies, South Africa, the Ural range and the U.S.A. (Table 4). Monazite sands are essentially orthophosphates of the rare earths, thorium and uranium; and the monazite is naturally associated with normal silica sands and gravel. Dressed monazite may contain as much as 15% thorium and monazite sands are capable of enrichment by magnetic separation and gravity concentration methods.

Extraction of thorium

The first stage in extracting thorium from monazite concentrates is the digestion of the sand to produce a water soluble product by the action of

TABLE 4 Typical composition of monazites %

	India	Brazil	U.S.A. (Idaho)
Total oxides	63.2	68.1	68.9
ThO ₂	9.4	6.5	3.95
CeO ₂	28.5	—	29.2
U ₃ O ₈	0.37	0.18	0.15
P ₂ O ₅	25.9	26.0	28.5

either hot concentrated sulphuric acid or hot concentrated sodium hydroxide solution.

Leaching with concentrated sulphuric acid at 200-230°C. for about four hours in autoclaves yields a pasty mass which is then diluted considerably with cold water. This gives a solution containing the bulk of the rare earth and thorium sulphates, collectively known as monazite sulphate which may be decanted from the coarse, undigested sand and fine silica sludge. The reaction as far as the thorium is concerned may be represented as $\text{Th}_3(\text{PO}_4)_4 + 6\text{H}_2\text{SO}_4 \rightarrow 3 \text{Th}(\text{SO}_4)_2 + 4\text{H}_3\text{PO}_4$.

The treatment of monazite sulphate solution to yield its components may be by precipitation after adjusting the pH, by fractional crystallization or by selective solvent extraction. If the pH of the solution is adjusted to about 1.0 by neutralizing with ammonia, a precipitate of thorium and rare earth phosphates and sulphates occurs which may be filtered off. This mixture may be redissolved and sparingly soluble thorium sulphate precipitated from a sulphate-excess solution which retains the relatively soluble rare earth sulphates.

Alternatively, the thorium concentrate may be redissolved in nitric acid and treated with tributyl phosphate as a selective solvent. This extracts the thorium compounds almost free from rare earth contamination. There are several variations of the solvent extraction method employing tributyl phosphate, depending on the starting solution, and whether the solvent is used as a first stage stripping process or as a refining process. However, the capital and labour costs of solvent extraction are generally attractive and most of the solvent can be recycled.

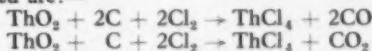
Other methods of selecting thorium compounds include precipitation of the rare earth carbonates from a neutralized acid solution leaving thorium in solution as a soluble complex, $\text{Na}_2\text{Th}(\text{CO}_3)_3$; precipitation of thorium iodate from strong nitric acid solution, precipitation of thorium oxalate from acid solution, and other special precipitates, these processes having limited commercial value.

If boiling concentrated caustic soda is used to leach monazite sands, the metal phosphates are converted to insoluble metal hydroxides and soluble trisodium phosphate. The reaction may be represented as $\text{Th}_3(\text{PO}_4)_4 + 12\text{NaOH} \rightarrow 3\text{ThO}_2 + 4\text{Na}_3\text{PO}_4 + 6\text{H}_2\text{O}$. The fine sand is treated for about three hours at 140°C. with 45% caustic soda. The insoluble hydroxides are filtered off hot and dissolved in hydrochloric acid or nitric acid and extraction of thorium from that solution follows similar lines to the methods used in the entirely acid extraction process.

The thorium recovery processes mentioned yield the thorium in various forms in solution or as a precipitated compound. Following this stage the

thorium is usually required in the oxide form. The purest oxide results from calcining thorium oxalate precipitated from a nitrite solution with oxalic acid but the latter is expensive. Precipitation of the hydroxide by ammonia, drying and igniting the precipitate again to thoria is an alternative, cheaper from the point of view of reagents but more difficult to filter. The cheapest and preferred route is evaporation of a nitrate solution and igniting the solid nitrate to oxide.

Non-hygroscopic double fluorides containing thorium can be prepared by wet chemical methods without going through the oxide stage. Thorium chloride, on the other hand, is generally prepared dry because it is hygroscopic and difficult to handle. Thoria is used and is chlorinated in the presence of carbon at 800-900°C. The most economical route at present appears to be the carbonization of a thoria/starch mixture at 850°C. to give a very intimate mixture of thoria and carbon which is then exposed to chlorine gas at 900°C. The reactions involved are:—



Reduction of thorium compounds

Thorium metal, because of its reactivity at elevated temperatures, particularly with the atmosphere and with containers, and its high melting point, 1,750°C., is difficult to isolate from the available compounds. Most of the extraction methods yield a powder or granular product and are followed by powder metallurgy processes to produce workable metal shapes.

In general, thorium can be prepared from thorium halides by reduction with reactive metals such as sodium, calcium and magnesium or by the electrolysis of a halide in a fused salt bath. Thorium oxide may also be reduced by calcium and, finally, thorium can be refined by the Van Arkel method which makes use of the reversible reaction of iodine with thorium in the vapour state.

Reduction processes. Calcium, magnesium and sodium are all feasible reductants for thorium chloride. The magnesium reduction offers certain advantages over the use of calcium for large-scale operations since it requires less magnesium per pound of product and magnesium is obtainable cheaper and purer than calcium. On the other hand the magnesium process requires a high quality thorium tetrachloride while the calcium process will tolerate higher oxygen contents in the tetrachloride for a given quality of thorium produced. The magnesium process tends to give rise to ThO_2 and ThOCl_2 contamination. The reduction processes are carried out in a pressure vessel and yield a powder or sponge metal product.

Thorium tetrachloride can also be electrolysed

in a fused salt bath to yield a granular thorium product. Sodium and potassium chloride eutectic is added to the electrolyte to depress the melting point. The operation is carried out in a carbon-lined vessel, the carbon acting also as the cathode, or in a silica vessel with a molybdenum cathode (fig. 10). During electrolysis, chlorine is liberated and this protects the melt, but during charging dry argon is used to protect against oxidation. The exclusion of oxygen and moisture is essential since thorium oxychloride gives rise to sludgy non-adherent thorium deposits of fine particle size, difficult to separate from the electrolyte and of high oxygen content. The cell is operated at 700-800°C. The thorium deposit consists of a cake of coarse thorium powder, chloride salts and fine thorium particles. It is agitated in water to leach out the salts, thus freeing the powder which is washed, separated and vacuum dried. The purity of thorium produced in this way is better than 99.8%, the major impurity being chlorine.

A fused electrolyte of thorium fluoride and sodium and potassium chloride also yields thorium in powder or granular form but thorium fluoride is usually reduced pyrometallurgically by calcium in the Ames process. This is a thermal reduction using a mixed charge of thorium fluoride, calcium metal and anhydrous zinc chloride. After ignition, by placing the vessel fully charged in a furnace, the reaction proceeds spontaneously to yield thorium, zinc, calcium chloride and calcium fluoride all in the molten state. In fact, the vapour pressure of zinc at the reaction temperature is high, necessitating the use of a closed pressure vessel lined with dolomitic or calcium oxide. The heat of reaction between the zinc chloride and calcium boosts the overall reaction and the liberated zinc alloys with thorium to give a lower melting point phase. The metal and slag readily separate and solidify conveniently in layers, the combination of calcium fluoride and chloride being fluid at reaction temperatures. The thorium is separated from the zinc, after separating the solidified metal and slag, by distilling off the zinc *in vacuo*. This yields thorium sponge which may be melted in a high-frequency furnace under vacuum and cast into a graphite mould, also under vacuum.

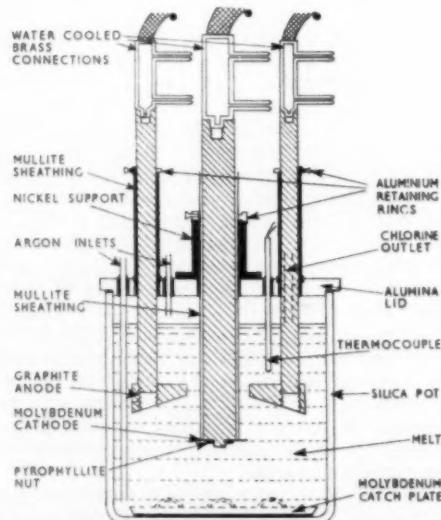
Methods of reducing thorium dioxide by calcium have been used for many years. Thoria, up to 50% excess calcium metal, and calcium chloride, are heated together in a closed steel pressure vessel for about 1 h. at 1,050°C. This yields thorium powder which may be released from the subsequently cooled and solid charge by leaching out the calcium oxide and chloride and excess calcium. The metal powder is washed and dried with the minimum exposure to oxidation and then compacted and sintered at 1,300°C. to give solid ductile thorium of 99.7%.

purity. The calcium chloride used in the reduction process acts as a flux and the amount used is fairly critical for a good yield of thorium unless a large excess of calcium is used.

Until the development of nuclear power technology few attempts had been made to extract thorium metal on anything but a very small scale. The early requirements of the nuclear power programme for thorium are still small and consequently it is not possible at this stage to predict the most economical route for the extraction of thorium from a given ore. Nevertheless, a variety of techniques have been developed in anticipation and it should not be difficult to combine various sections into an economical whole.

Refining and casting thorium

Bomb reduced thorium may be compacted and vacuum melted in a thoria or beryllia crucible giving marked reductions in oxygen, hydrogen and nitrogen contents and more homogeneity. The hydrogen is pumped off as the result of hydride decomposition and oxides and nitrides tend to be floated out of the melt. If a graphite mould is used for casting, slight surface contamination of thorium carbide results on the ingot. This may be removed by pickling with dilute nitric acid and sodium silico-fluoride solution. The high melting point of thorium and its reactivity towards refractories presents a number of melting problems. Alumina, magnesia and lime are attacked rapidly by molten thorium. Thoria is resistant but tends to contaminate the melt with oxide and is very susceptible



19 Thorium electrolysis cell

to thermal shock. Zirconia has been successfully used but is increasingly attacked as the crucible temperature exceeds the melting point of thorium. Beryllia has been found to be very satisfactory in that it has a comparatively good thermal shock resistance and adequate strength at high temperatures. It resists attack by molten thorium at 1,800°C. and may be used for short periods at 1,950°C.

For laboratory melting the use of a tungsten arc directed on thorium on a water-cooled copper hearth under an atmosphere of helium or argon is suitable. This method is limited by the small amount of metal which may be loaded into the furnace and melted into one pool; a matter of 100 g. or so. The extension of this method to the use of consumable electrode arc melting is applicable to thorium on a much larger scale. In this method thorium sponge or other suitable material is fabricated into an electrode which replaces the inert tungsten electrode and this is fed gradually into the furnace holding the inert atmosphere. The disadvantages of the arc method are that at inert atmosphere pressures low enough to get effective degassing and appreciable refining, the arc is unstable; additionally the melting effect is very localized so that homogeneous melting is impossible. The latter difficulty may be overcome to some extent by melting the first consumable electrode to form an electrode shape which can be remelted to form an ingot.

Finely divided thorium or massive thorium cut into chips may be refined by the iodide, Van Arkel process. This is a small-scale batch method very similar to that described for uranium. Thorium tetrailode, produced as a vapour by the reaction of iodine vapour on the small thorium pieces or particles, is thermally decomposed on a hot thorium wire filament to produce thorium of a very high

grade, containing 0.02% carbon and less than 0.01% of oxygen and nitrogen. The reaction between thorium and iodine takes place at 400°C., and is reversed near the electrically heated hot wire at a temperature of 1,700°C. The iodine released is able to repeat the cycle. The major difficulty in extending the scale of this process is in keeping the wire at temperature as its cross-section builds up as the thorium deposits. The reaction vessel is normally made from heat-resistant glass and a typical starting size for the filament would be 115 cm. long by 0.64 mm. dia. Before starting the reaction the complete charged apparatus has to be outgassed for some 24 h. at 500°C., during which time the iodine is refrigerated.

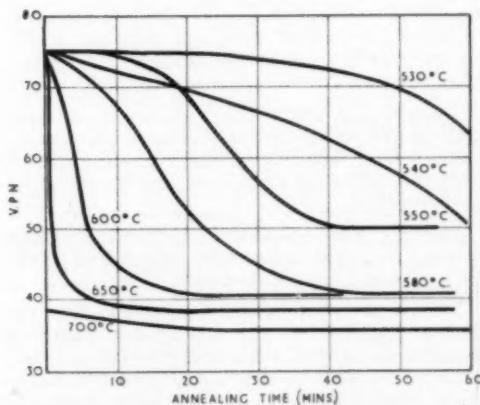
Fabrication of thorium

The amount of work required to deform thorium mechanically even in the cold state is not great in comparison with most metals, and thorium can be hot or cold formed readily in most operations. It is possible, for instance, to cold roll thorium to give a reduction in excess of 99% without intermediate annealing, provided the thorium is sufficiently pure. With impurities and alloying additions more work is required in deformation but the processes are still generally applicable. Even thorium containing oxygen to a level of 5-6% has considerable ductility because the oxides are formed as small inclusions which do not seriously impede metal flow. Impurities segregating at grain boundaries in a more irregular form do introduce some difficulties in working.

In relatively massive hot working operations, such as forging and extrusion, thorium may be heated without special protection to about 800°C. and the working is relatively easy. Hot working or heat treatment of small pieces, particularly if they have a high surface area to volume ratio, will generally require some atmosphere protection for the metal. Preheating for hot rolling may be done in a lead bath at 600-700°C., for instance.

Cold rolling of thorium sheet tends to produce rather marked preferred orientation, most of which is removed by recrystallization. Preferred orientation is also evident in extruded stock and the type and extent of preferred orientation is to a large extent dependent on the rate of extrusion. At slow extrusion speeds the orientation is not as simple as that resulting from fast extrusion rates and slowly extruded thorium tends to be considerably stronger than fast extruded stock.

On annealing severely cold-worked unalloyed thorium, softening becomes evident at 500°C. or slightly lower. At 600°C. softening is complete after 20-30 min. and, as with other metals, the temperature at which softening of cold-worked



11 Recrystallization curves for iodide thorium

thorium begins decreases as the degree of previous cold work increases (fig. 11).

Because thorium has a marked tendency to stick to die surfaces, operations like wire and tube drawing and swaging present a little trouble. It is possible to alleviate this by sheathing or plating the thorium with a metal such as copper which has very little tendency to weld into the die.

The machining of thorium can be by conventional methods, wet or dry, but the softness of the metal makes the production of a fine finish difficult. Machining is generally carried out at slow speeds and with heavy cuts as when machining magnesium and its alloys. The heavy chips produced are less liable to spontaneous ignition than fine swarf.

Powder metallurgy of thorium

Several processes for producing thorium yield the metal in powder or granular form. Thorium in the form of sponge, chips or turnings can be converted to powder by the controlled formation and decomposition of the hydride. Although there are

two hydrides of thorium, ThH_2 and Th_4H_{15} , it is the dihydride which is used for this purpose. It is formed by reaction of thorium in a hydrogen atmosphere at about 650°C . This hydride is then decomposed in vacuum at about 750°C . and forms a sinter cake which may be pulverized to a serviceable powder. In general, particles of high purity thorium metal are quite malleable and the powder is readily compacted and sintered into ductile shapes.

Fine thorium powder and thorium hydride are pyrophoric, particularly when moist. Heat evolved by reaction with air and moisture is capable of igniting evolved hydrogen. Material for storage is therefore protected, simply by collecting it under a non-reactive low boiling point liquid. When extensive handling has to be carried out this is done in an inert atmosphere, conveniently in a glove box. Sintering is also carried out in an inert atmosphere or in a vacuum. In the simple compacting followed by sintering process, the compacting pressure will be about 20 tons and the sintering temperature $1,400^\circ\text{C}$.

Induction hardening

AN automatic induction hardening installation is now in use at the Edmonton works of British Oxygen Engineering Ltd. At present employed for case-hardening short shafts and through-hardening bushes, the installation will eventually be used on a much wider range of components.

Supplied to British Oxygen Engineering by Delapena and Son Ltd., of Cheltenham, the equipment minimizes distortion and lowers the cost of hardening. Since only the surface layers have to be heated, there are substantial economies in power consumption. Scaling is virtually eliminated as quenching can be carried out immediately after heating.

With the new installation, application of heat can be precisely controlled, and the hardening process is rendered clean and fast. Heat is generated only in the part being treated and heat losses during standby periods are eliminated. After a period of close-down the full power output is available within three minutes.

The equipment consists of an induction heater; a vertical shaft hardening machine with special fitments enabling it to be used for drop quench operations; a radio-frequency transformer, and inductor coils for heating a wide range of components; a water circulating system to ensure adequate cooling of the induction heater, transformer and

induction coils, and sufficient flow for spray quenching.

Operating sequence

The shaft hardening machine can handle shafts up to 4 ft. long. The shaft is held vertically between adjustable centres, the lower one being driven at a variable speed by an electric motor. The shaft is heated by a surrounding inductor coil which moves upwards over the length to be treated. The rates and distances of traverse are variable and the machine can be set to cover parts of a shaft at different speeds. At the beginning of each heating period the inductor coil is held stationary until the correct temperature is reached, the duration of these pre-heating periods being separately controllable.

Quenching is performed continuously by water sprayed from a ring mounted just below the inductor coil. Rate of flow is controllable and the quench continues until a pre-set interval after the end of the heating period.

Once set up for a particular job, the whole process is automatic. The operator has only to load a shaft by hand and press the starting button.

To use the shaft hardening machine for drop quench applications, the transformer carrier is removed and a small work table fitted in its place, while an auxiliary quench tank is placed on top of the normal drain tank. As with shaft hardening, the whole sequence is automatic.

Prespheroidizing

Modification of the cast structure of high-speed steel

HIGH-SPEED STEEL is the most popular material for the manufacture of cutting tools. Its unique properties are conferred by the presence of substantial quantities of alloying elements, notably tungsten. Some typical compositions are:

Type	C	W	Cr	V	Mo	Co
18-4-1	0.7	18.0	4.0	1.0	—	—
6-5-2	0.85	6.0	4.0	2.0	5.0	—
18-4-1+Co	0.75	18.0	4.5	1.25	—	5.0
9-3-3-8	1.33	9.0	4.25	3.5	3.2	8.5

Because of the particular composition of high-speed steel, the freezing process is extremely complex and gives a structure like that shown in fig. 1. There are large colonies of eutectic, and the structure is by no means homogeneous.

This structure is normally not acceptable for tools, and has to be broken down by hot work: forging, rolling or extrusion. The progress of this break-up is shown in fig. 2; the eutectic colonies, fig. 1, are elongated into stringers of carbide, but the structure remains heterogeneous. The presence of gross eutectic colonies can cause the steel to break up during hot work.

The stringers are generally considered to be undesirable; they can cause distortion or failure during heat treatment, and have been blamed for countless failures of high-speed steel tools in service.

Until recently it was believed that heat treatment alone could not alter the cast structure, fig. 1. However, work in BISRA has shown that it can. The irregularities in fig. 1 are thought to be caused by lack of equilibrium; that is, in the freezing operation there was insufficient time for completion of a peritectic reaction. If this reaction were allowed to proceed, it should, it was reasoned, give a structure free from eutectic, and should therefore eliminate the coarse stringers.

The technique adopted is to re-heat the as-cast steel to the reaction temperature (about 1,320°C.), at which the eutectic actually becomes molten. But the steel is then in the same state as it was just before the ingot froze, and by holding it at this temperature reaction takes place between liquid and solid to give a new solid phase without

eutectic. This operation has been termed prespheroidizing, because it enables the carbide (eutectic) to be spheroidized, or broken up into small globules, before any hot work is carried out.

The effect of prespheroidizing on an ingot structure is shown in fig. 3.

After hot working the structure becomes comparatively uniform with an almost complete absence of stringers.

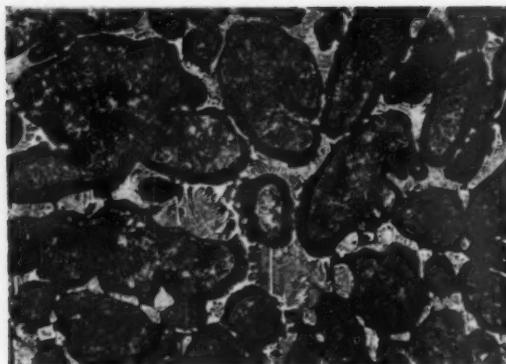
It must be emphasized that the process is not yet a commercial proposition. The practical difficulties involved in the treatment of large masses of metal at the temperatures involved are great. An accidental temperature rise of as little as 5°C. could be disastrous, and the metal might melt sufficiently to lose its shape and be completely ruined. Also it has yet to be established that the treatment improves the properties of high-speed steel, and if it does whether the improvement is sufficient to warrant the expense involved. The carbide particles in prespheroidized samples are larger than is usually considered desirable and this may have an embrittling effect.

Possible applications

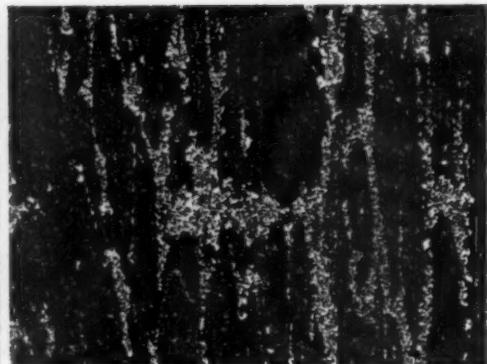
There are two applications, however, where prespheroidizing could be of immediate value. A small proportion of high-speed steel tools are precision-cast to shape, and are heat treated without any hot work being carried out. These tools contain eutectic, but because of the small size of the section the structure is generally finer than in ingots, fig. 5. The prespheroidizing temperature is only about 30°C. above the normal hardening temperature for high-speed steel, so that in this case the tool can be prespheroidized, quenched, and tempered, and is then ready for final grinding and use. The masses involved are small, and this means that temperature control is much easier—either using a muffle furnace or, preferably, a salt bath. Handling is also much simpler. Fig. 6 shows the prespheroidized structure.

A further application is the restoration of overheated tools. If the correct hardening temperature is exceeded, the structure will revert to a eutectic structure. Such tools can be prespheroidized in the same way as cast cutters and an expensive rejection may be avoided.

This summary is based on BISRA reports MG/L/197/58, 'The reduction of carbide heterogeneity in high-speed steel by thermal treatment of the ingot,' by G. Hoyle and E. Ineson, MG/L/44/59, 'An investigation of the prespheroidizing process by thermal analysis,' by A. Hollingsworth and G. Hoyle, and MG/L/50/59, 'The reduction of carbide heterogeneity in high-speed steel by thermal treatment of the ingot: further experimental results,' by D. B. Hammond and G. Hoyle.

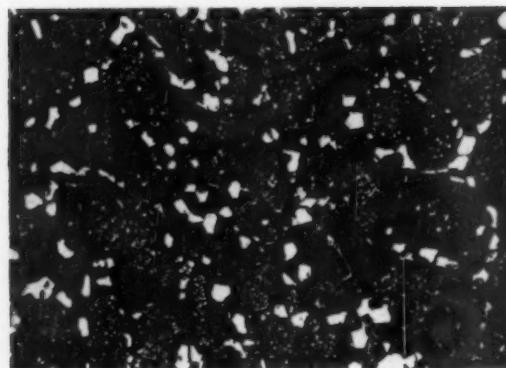


1 High-speed steel ingot—*as cast*



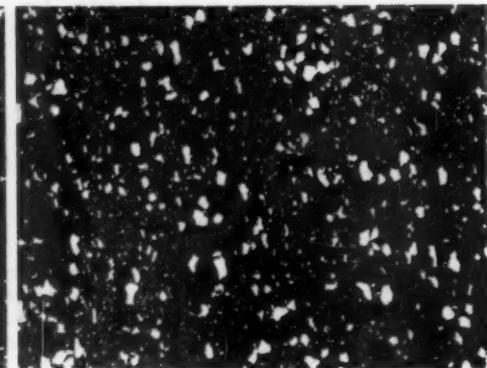
2 Two-inch billet forged from ingot

$\times 100$

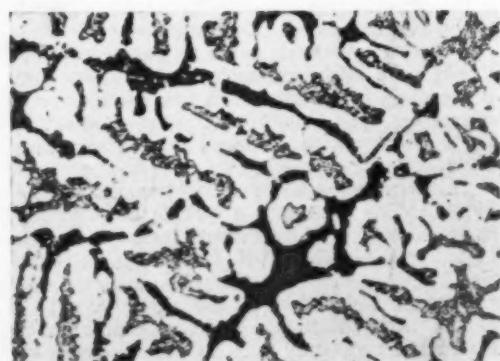


3 High-speed steel ingot—*prespheroidized*

$\times 100$

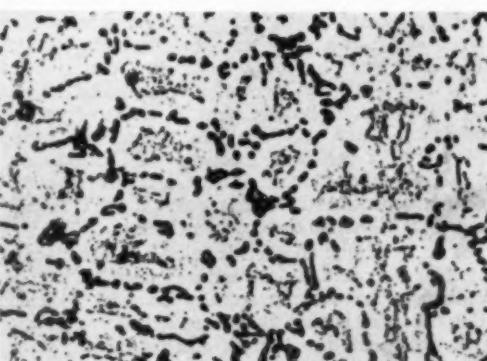


4 Two-inch billet forged from prespheroidized ingot $\times 100$



5 Cast cutter—*untreated*

$\times 500$



6 Cast cutter—*prespheroidized*

$\times 500$

Application of electron microscopy

Investigation into the subgrain structure of high-purity aluminium

DR. H. BICHSEL

The subgrain structure of roll-hardened and annealed high-purity aluminium foil has been investigated by transmission electron microscopy. It is shown that a cell structure arising after polishing comes from local etch pits and is not directly connected with the subgrain structure. The author of the following article, the German version of which appeared in 'Metall,' March, 1960, is with the Aluminum-Industrie-Aktien-Gesellschaft Research Laboratories at Neuhausen am Rheinfall, Switzerland.

IN RECENT YEARS great strides have been made in the sphere of metallurgy through the application of electron microscopy. Above all it has proved possible to throw light on the problem of lattice faults (dislocations) and their effects on the properties of metals. Numerous investigations have also been carried out concerning substructures in crystals.

One way in which it is possible to produce subgrains consists in subjecting lightly worked metal to annealing at a high temperature, at 500–600°C. for instance. This method is used especially by the French school and by R. W. Cahn¹ in his fundamental work on polygonization. C. Crussard² designates the process which takes place as recrystallization *in situ*, since the resultant subgrains, which can display considerable sizes of 20–1,000 μ , adopt almost the same orientation.

Subgrains also arise after heavy cold working and subsequent annealing to remove strain hardening; in this instance the degree of deformation, the annealing conditions and the purity of the metal determine their size. In general it may be said that the subgrains are correspondingly smaller, the higher is the degree of deformation, and correspondingly larger, the higher is the annealing temperature and the greater the degree of purity. Likewise the temperature at which the cold working is carried out has an influence on the size of the subgrains. Thus aluminium hammered in liquid air shows only an imperfect, and clearly smaller, substructure than metal deformed at room temperature.³ Furthermore, substructures are also formed during hot working.

P. B. Hirsch⁴ gives a comprehensive picture of the most important works published on substructures (mosaic structures) up to 1956.

Substructures in crystals can be identified either by X-ray methods, or by either optical or electron microscopy. It is, above all, the improved micro-X-ray methods which have produced evidence of these subregions, which show only extremely small differences in orientation.^{5, 6} Dependent on the experimental conditions, in highly deformed aluminium subgrains of the order of a few microns are to be found. By means of a fine radiation X-ray method, P. B. Hirsch and N. J. Kellar observed subgrains of a size 1–2 μ in pure aluminium (99.999%) after 57% vertical deformation by rolling.

Direct metallographic evidence of the subgrain structure is rendered difficult by the fact that the low-angle grain boundaries cannot in practice be etched. This may mainly be attributed to their low energy. In addition, according to G. Wyon and M. J. Marchin,⁷ dislocations in aluminium can only be made visible by etching, if they show a Cottrell atmosphere. P. A. Beck and Hu Hsun,⁸ and also E. C. W. Perryman,⁹ investigated the deformed areas in pure aluminium in polarized light; the latter author also made use of phase contrast illumination. This process is based on the fact that the small differences in the orientation of the base metal are also present in the optically anisotropic, oxide layer, and in polarized light in the presence of a compensator plate appear as colour shades. Nevertheless only vaguely defined

areas, which gave certain indications of the deformation process, could be observed, but not the actual substructures themselves.

More recently the electron microscope is now being applied with great success to the investigation of the fine grain structure of metals. As investigation techniques, two methods may be employed: either a surface print of the object is produced, or on the other hand the object (the metal foil) is thinned down by means of special methods to less than 0.1μ , so that it is suitable for direct transmission. In particular the last method has yielded a large number of fundamentally novel results. Not only may substructures be observed in a simple way, but lattice faults and their arrangement can be recognized. The first of these works was produced by R. D. Heidenreich,¹⁰ who found substructures of the order of $1-3 \mu$ in size in cold-worked aluminium. Since, according to the theory of electron diffraction, the intensity of the electron beam transmitted during passage through a crystal is dependent on the orientation of the lattice, the subgrains appear as zones of differing luminosity. At the same time even the smallest differences in orientation, such as occur with subgrains, are recorded.

Areas with high local stresses (dislocations) absorb the radiation to a very great extent. P. B. Hirsch, R. W. Horne, and M. J. Whelan,¹¹ investigated the distribution and movement of dislocations in beaten aluminium foils. During observation in the electron microscope the dislocations move in consequence of the slight heating up of the specimen by the electron beam. Recently the behaviour of dislocations under tensile load has been described. A device constructed by H. G. F. Wilsdorf¹² made possible the extension of thin metal foils directly under observation. From these investigations the conclusion must at all events be drawn that the mechanism of deformation in thin foils takes place in a different manner than in bulk material. Finally subgrains can be revealed in alloys, e.g. Al-4% Cu, since the alloying component is preferentially precipitated at lattice



2 Subgrain structure of high purity aluminium foil, roll hardened. (Transmission micrograph)

defects after solution annealing and tempering.^{13, 14, 15}

In the present work a report is given of observations made by means of transmission microscopy during the growth of subgrains as a result of various annealing treatments of thinned metal foils, and of investigations of the cell structure arising after chemical polishing of aluminium in the Alcoa R-5 bath.

Experimental section

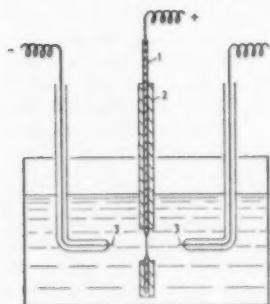
Growth of subgrains in high purity aluminium foil after heat treatment

As initial material use was made of roll-hardened, pure aluminium foil, 30μ thick, of the following chemical composition:

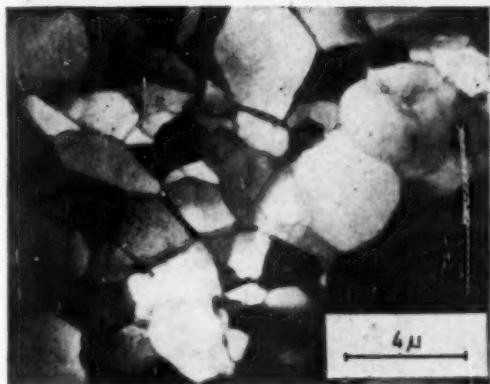
Si 0.0046%	Cu 0.0006%
Fe 0.0010%	Al 99.9938%

Sections of the foil were annealed at 100, 160, 200, 210 and 250°C , in each instance for 24 h. Subsequently the foils were thinned down in accordance with the method of W. Bollmann¹⁶ with the use of a tribasic sodium phosphate solution (160 g./litre) as electrolyte (fig. 1). Oxide films adhering to the specimen were dissolved by careful immersion of the foil for a period of 1 min. at $90-100^{\circ}\text{C}$. in Smudge Remover Solution (100 cm.³ phosphoric acid d = 1.7, 100 g. chromium trioxide and 400 cm.³ water). After thorough washing in water the foil is transferred into alcohol. The thin metal tinsel fragments can subsequently be withdrawn on carrier grids.

Fig. 2 shows the substructure of the roll-hardened foil. Sharply defined regions of the order of about 2μ in size can be recognized. In the interior the subgrains often show dark lines which end on the boundaries of the subgrains. These are the extinction contours which have already been described by R. D. Heidenreich,¹⁰ which arise either through



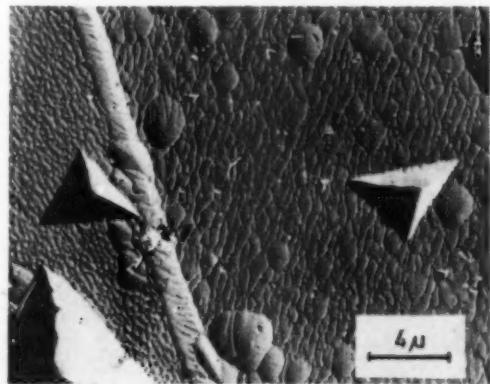
1 Bollmann apparatus for the production of thin metal specimens suitable for transmission electron microscopy.
1, Aluminium foil.
2, Resin coating.
3, Pt electrodes



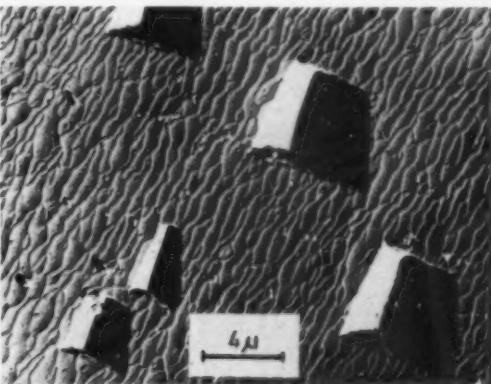
3 Subgrain structure of high purity aluminium foil, annealed 24 hours at 200° C. (Transmission micrograph)



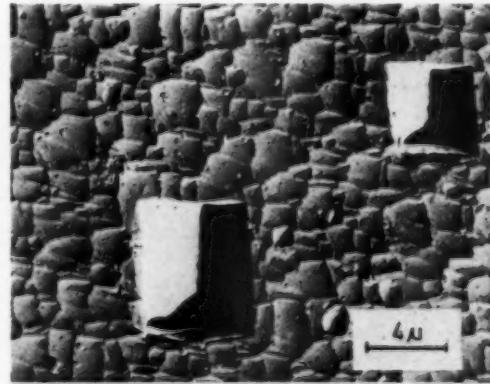
4 Subgrain structure of high purity aluminium foil, annealed 24 hours at 200° C. Marked growth of individual subgrains. (Transmission micrograph)



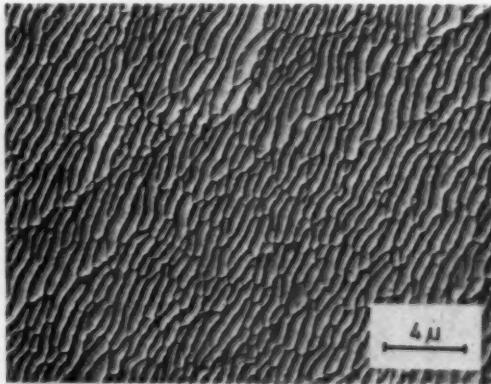
6 High purity aluminium sheet, soft annealed. Electrolytically polished and finally polished for 2 minutes at 90° C. in the Alcoa R-5 Bright Dip Bath. (Carbon replica)



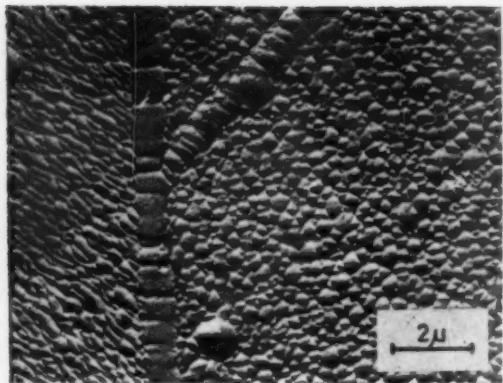
7 High purity aluminium sheet, soft annealed. Electrolytically polished and finally polished for 2 minutes at 90° C. in the Alcoa R-5 Bright Dip Bath. Orientation close to (110). (Carbon replica)



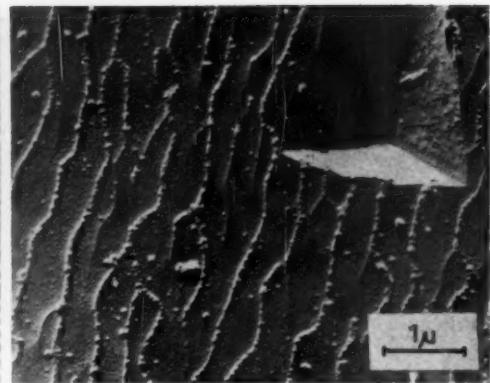
8 High purity aluminium sheet, soft annealed. Electrolytically polished and finally polished for 2 minutes at 90° C. in Alcoa R-5 Bright Dip Bath. Orientation close to (100). (Carbon replica)



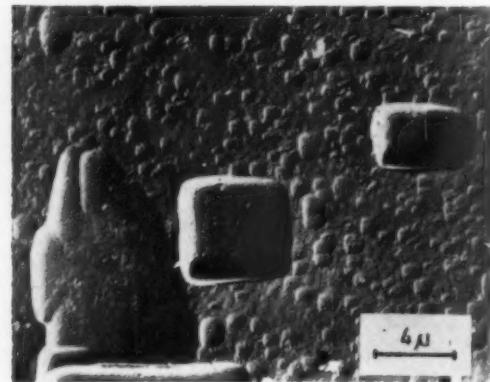
9 High purity aluminium sheet, 70% reduction by rolling. Polished for 3 minutes at 90°C. in Alcoa R-5 Bright Dip Bath. (Carbon replica)



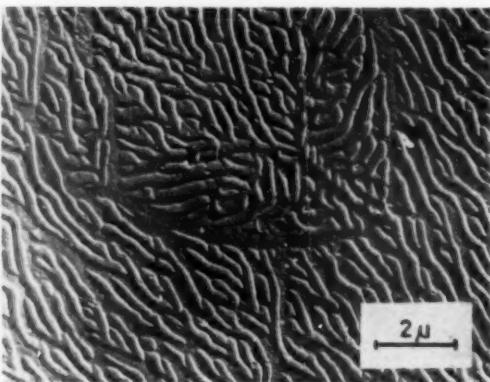
10 High purity aluminium plate, soft annealed and lightly worked. Electrolytically polished and finally polished for 2 minutes at 90°C. in Alcoa R-5 Bright Dip Bath. Slip band (oblique line) ending on a grain boundary (vertical line). (Carbon replica)



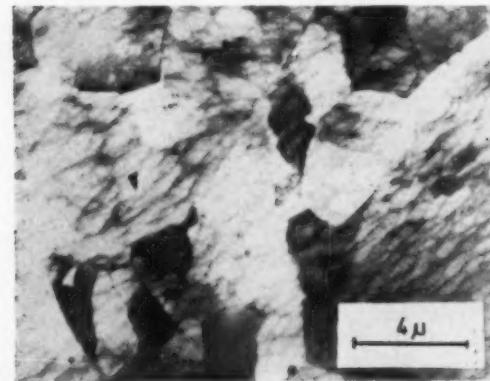
11 High purity aluminium sheet, soft annealed. Electrolytically polished and finally polished for 2 minutes at 90°C. in Alcoa R-5 Bright Dip Bath. Deposit on the cell ribs. (Carbon replica)



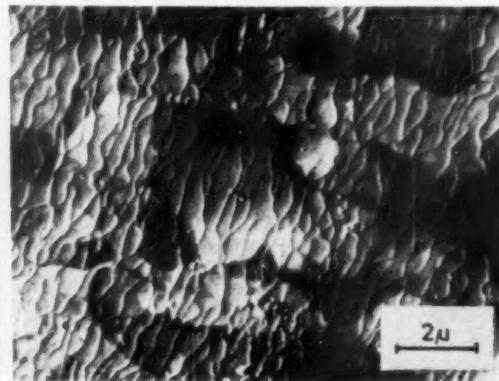
12 High purity aluminium sheet, soft annealed. Polished for 30 seconds in Alcoa R-5 Bright Dip Bath. Orientation close to (100). (Carbon replica)



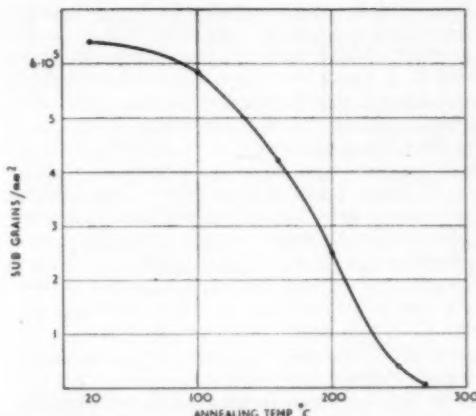
13 High purity aluminium plate, soft annealed. Electrolytically polished and finally polished for 15 minutes at 90°C. in Alcoa R-5 Bright Dip Bath. (Carbon replica)



14a High purity aluminium foil, annealed for 24 hours at 200°C., and thinned down in Alcoa R-5 Bright Dip Bath. Not shadowed with chromium. (Transmission micrograph)



15 High purity aluminium foil, annealed for 24 hours at 130°C. and thinned down in Alcoa R-5 Bright Dip Bath. Strongly shadowed with chromium. (Transmission micrograph)



5 Growth of subgrains in roll hardened high purity aluminium foil after various annealing treatments. Annealing period 24 h.

variations in thickness or during slight bending of the foil. Alongside these lines dislocations can also be seen. These remain unchanged during the bending of the specimen by 1-2°, whereas the interference lines are extinguished.

After annealing of the strips at the temperatures already mentioned, a continuous coarsening of the subgrain structure takes place, as is shown by fig. 3. Even after 24 h. annealing at 200°C. the grains of the structure become disproportionate in size, for a few individual grains grow to be extremely large (fig. 4). These coarse grains which absorb the smaller ones, finally grow into the primary, recrystallized grain structure.

By means of photographs the subgrains can be counted. Table 1 and fig. 5 contain the average calculated numbers of subgrains per mm.² From the X-ray diffraction diagram the degree of recrystallization may be assessed in each instance.

The value \bar{d} corresponds to the average subgrain diameter at the relevant annealing temperature,

on the assumption that the sublattices are cubic with an edge length \bar{d} .

Between 20 and 100°C. no noteworthy change in the subgrain structure takes place. From 100°C. onwards, however, a marked reduction in the number of subgrains occurs. In this temperature range the breakdown of the greater part of the strain hardening probably takes place.

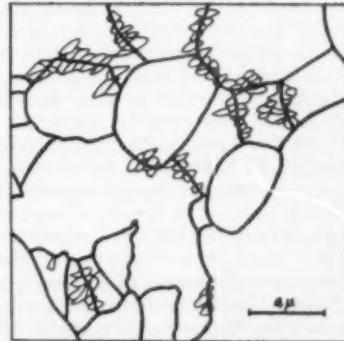
The origin of subgrains is still relatively obscure. Originally the polygonization process was assumed to be the most important mechanism. After heavy working, however, in consequence of the simultaneous activity of several slip systems fundamentally more complicated conditions exist, than during classical experiments with polygonization. Through the formation of Lomer-Cottrell barriers¹⁷ the movement of the dislocations is retarded, which makes itself apparent through the increase in the strain hardening. Where blocked dislocations occur, however, the course of polygonization is impeded. Very recent electron microscope investigations of thinned nickel foils carried out by W. Bollmann¹⁸ showed that at points of lower dislocation density nuclei are formed, from which subgrains arise, capable of growth. In this process the critical size of nucleus at which growth is possible is dependent on the local distribution of the dislocations.

The results obtained in the present investigation of the subgrain structure in the roll-hardened state and after various annealing treatments give rise to the assumption that the growth of the structure from the subgrain up to the very coarse primary grain is a process of continuous evolution in high purity aluminium. In this sequence the largest subgrains present in the rolled state, in accordance with the lowest surface tension, should have the best opportunities for growth. In the course of the growth of the subgrain the selective growth already described by P. A. Beck and col-

TABLE 1 Subgrain and recrystalline data

Annealing treatment	Subgrains/mm. ²	\bar{d} (μ)	Degree of recrystallization
Roll hardened	640,000	1.25	Not recrystallized
100°C./24 hours	585,000	1.31	" "
160°C./24 "	420,000	1.54	Start of recrystallization
200°C./24 "	250,000	2.00	ca. 5-10% recrystallized
250°C./24 "	38,000	5.10	ca. 50-60% recrystallized
270°C./24 "	ca. 500*	ca. 45*	Completely recrystallized

*Primary grain determined by means of an optical microscope.



14b Superimposition of the cell structure on the subgrain boundaries (cf. fig. 14a)

laborators¹⁹ comes into operation, whereby those grains grow the fastest which display favourable mutual orientations in relation to their neighbours. Small grain boundary movements in unit time produce neighbouring grains with similar orientation or in spinel twinning position.

Connection between surface structure produced by means of a chemical polishing bath and the lattice substructure

After treatment of aluminium surfaces in polishing baths of the phosphoric acid-nitric acid type (e.g. Alcoa R-5 Bright Dip = 470 cm.³ phosphoric acid d = 1.7, 30 cm.³ conc. nitric acid, 150 cm.³ distilled water) regular cell structures arise, which can readily be revealed by electron microscopy. M. S. Hunter and D. L. Robinson,²⁰ who published a first study of the subject, as well as D. Altenpohl and W. Hugi,²¹ are of the opinion that through the specific attack of the polishing bath the subgrain structure is brought to light. The dimensions of the cell structure, as has also been confirmed later by F. B. Cuff and N. J. Grant,²² are dependent on the purity of the metal; the purer the metal the larger are the cells.

Fig. 6 shows the surface of a soft-annealed, pure aluminium sheet after treatment in the Alcoa R-5 polishing bath. The photograph shows two essential things, the form of the individual cells and their dependence on the orientation of the grain. In order to be able to determine the latter, after polishing, sheet specimens were etched with a solution of C. S. Barrett and L. M. Levenson²³ (9 parts conc. HCl, 3 parts conc. nitric acid, 2 parts 40% H₂F₂, 5 parts water), which reveals the (100) planes and produces cubic etch pits. For the electron microscope investigation carbon replicas were produced and shadowed obliquely with chromium at an angle of 30 deg. By means of the direction of the shadows, of the light-coloured flecks in the grain boundary of fig. 6, for instance, it is possible to show that the cell boundaries stand out in relief like ribs, since in each instance their light side is turned towards the 'light source.' This should be borne in mind, since during observation the structure often appears to stand out plastically in the same way as the grain, which leads to a false impression, and also misled M. S. Hunter and D. L. Robinson to the conclusion that the 'subgrain boundaries' would be etched by the R-5 polishing bath.

D. Altenpohl and W. Hugi,^{21, 24} as well as F. B. Cuff and N. J. Grant,²² on the other hand recognized that the cell boundaries are in relief and represent a system of ribs. These authors assume that traces of heavy metal (Fe, Cu) are preferentially piled up in the 'subgrain boundaries,' which are therefore nobler than the 'subgrain centre.' The polishing

bath, in their opinion, attacks the 'subgrain centre' first of all, whereas the nobler 'boundaries' remain in relief. This hypothesis, according to F. B. Cuff and N. J. Grant,²² is supported by the observation that the cell size is dependent on the purity of the metal. Similar cell structures also appear, as the numerous experiments of N. C. Welsh²⁵ show, after the use of various electrolytic polishing methods. N. C. Welsh found that the form of the cell structures, quite apart from the crystallographic orientation, is also dependent on the composition of the electrolyte and the polishing conditions. Thus for instance the cell size increases with the increase in the electrolysis voltage. From this behaviour N. C. Welsh draws the conclusion that the cell structures obtained could not be a matter of the actual subgrain structure.

As figs. 6, 7 and 8 show, the cell structure shows a marked dependence on the orientation of the grain. Especially typical structures result, if the intersected plane happens to be a (111), (110) or (100) plane. In the first instance a dotted structure is produced (left grain in fig. 6). On the (110) plane the cells appear elongated, and in the ideal instance parallel furrows are to be found, whereas on the (100) plane a pattern consisting roughly of squares occurs. These relatively regular distributions are wiped out with increasing cold deformation. Fig. 9 shows the surface of a cold rolled, pure aluminium sheet where the degree of reduction is 70%. The course of two parallel slip bands are clearly recognizable. In other instances it was found that, dependent on the orientation, the slip bands are attacked just as strongly as the grain boundaries. In fig. 10 the vertical line, as follows from the different cell structure on both sides of the line, is a grain boundary, whereas at about 45 deg. to it there runs a slip band, produced when the specimen was lightly worked. Often the cell ribs display deposits, which presumably consist of oxides (fig. 11).

The way in which the attack of the polishing bath starts after a short period of action (30 sec.), is clearly evident from fig. 12. First of all isolated, individual pits are formed, which show a certain similarity to the cubic etch patterns previously produced. During subsequent polishing treatment the pits increase in size, and grow flat in accordance with the action of the polishing bath. At points where two neighbouring pits meet, elevations, i.e. ribs of the cell structure, arise. After a very long period of action by the polishing bath the dependence of the cell structure on the crystalline orientation disappears to some extent, the cell ribs become wider, and round cells of irregular shape then form. In fig. 13 we have an etch pit with approximately (111) orientation, which after 15 min. action by the bath has again been levelled out.

Transmission microscopy of thin metal foils

afforded, in simple and unambiguous form, evidence that no direct connection exists between the actual subgrain structure and the cell structure disclosed above.

For this purpose the thin metal specimens were prepared in the following manner: high purity aluminium foil, 30 μ thick, was treated for a few minutes in the R-5 bath at 90°C., in order to bring out the cell structure on both sides. After washing and drying one side of the foil was covered with resin (polystyrene in trichloroethylene). After drying of the resin, the foil was again carefully thinned down in the R-5 bath, until holes and thin areas occurred. At this moment the treatment was broken off, and the foil washed off. Subsequently the layer of resin was removed by immersion in benzene, and the residual high purity aluminium foil was washed clean in benzene and alcohol.

During transmission microscopy, apart from the subgrain structure, the cell structure is also apparent; it may readily be recognized in fig. 14 (a). The impression is partly somewhat blurred, since the structures on both sides of the foil are visible at the same time. In fig. 14 (b) for the sake of better illustration the superimposition of the two structures is represented. Since the cell structure is formed in relief, *i.e.* the ribs are raised in relation to the interior of the cell, it can be brought out in greater contrast on thin metal foils also, by means of oblique vaporization with chromium (fig. 15). From the two last illustrations it is undoubtedly clear that in fact the cells do form a surface structure. In fig. 14 (a), without chromium shadowing, the ribs appeared darker than the background, since in consequence of the greater thickness of the specimen at this point more electrons were absorbed. It may be clearly recognized that no direct relationship exists between the cell structure and the sublattice structure. The cell structures extend without interruption right over the subgrain boundaries and are elongated, whereas the subgrains have a tendency towards polygonal form.

After the completion of these investigations a similar work of R. Phillips and N. C. Welsh²⁶ came to our knowledge. By transmission microscopy of thin foils, the authors investigated a surface structure set up by 1% hydrofluoric acid, which shows a certain similarity with that which has been described. The authors named also come to the conclusion that there is no connection between surface structure and subgrain structure.

Discussion

By means of transmission electron microscopy of thin foils of high purity aluminium, it proved possible to determine the size of the subgrains of

roll-hardened and lightly annealed material. Recently B. G. Ricketts, A. Kelly, and P. A. Beck,²⁷ at the AIME 1959 Fall Meeting, reported on the subgrain size of a rolled and annealed, high purity aluminium crystal. The average subgrain size of 80% rolled material, which was also found by means of transmission electron microscopy, was determined at 1.2 μ , which is in good agreement with our own measurements (1.25 μ). On the other hand these authors also found that after annealing at 300–400°C. the sublattice size of 2.7 μ was not exceeded. It must be taken into consideration, however, that the high purity aluminium crystal was rolled in the (110) [112] position, from which one may expect a very marked and sharp rolling texture. This has the result that between the individual subgrains in practice only low angle grain boundaries occur, for which according to P. A. Beck and collaborators¹⁹ the grain boundary movement velocity is very small.

The nature of the growth of the subgrains, starting from the roll-hardened state, seems at first to be a continuous process. After annealing at 160°C., individual subgrains, which are clearly in a preferential position in relation to their environment, start to grow more rapidly. The subgrain structure of foil annealed at 200°C. shows a certain similarity with the macrostructure of high purity aluminium sheet which has been annealed for some hours at 500°C. Here again some large grains appear, which have increased in size on account of their favourable conditions of orientation.

Transmission microscopy of thin metal foils produced unambiguous evidence that the surface cell structure arises as a result of the polishing process, and that there is no direct connection between it and the actual subgrain structure. The assumption that these cells are identical with the subgrains is contradicted by the finding that the cells, in contrast to the real subgrains, remain essentially unchanged during the course of an annealing treatment. The cell structures display differences only between the deformed and the recrystallized material. This seems to us to be an indication that lattice defects which produce a preferential, local etch attack contribute to the formation of the cell structure. This assumption is in agreement with the finding that the high angle grain boundaries and slip bands, in other words areas with high imperfection densities, are strongly attacked by the polishing bath (figs. 6 and 10). The wide strip, devoid of texture, in the grain boundaries, which is mentioned by F. B. Cuff and N. J. Grant,²² must well be none other than the result of a strong attack, which removes the metal along the grain boundaries to leave a pattern of furrows.

It is very probable that during chemical polishing, in consequence of the lack of an external, electro-

motive force, there is occurrence of local corrosion, brought about by the imperfections and impurities. In the latter instance we automatically have an explanation for the observation that the cell size increases with the rise in the purity of the metal. Likewise the strong attack on the grain boundaries can be thus explained.

In this connection a further consideration is likewise of interest: according to the determination of M. S. Hunter and D. L. Robinson²⁰ the number of cells in soft annealed material varies between 10^6 and $1.3 \times 10^7/\text{mm.}^2$ which corresponds to a mean diameter of $0.2-1.0 \mu$. For the assumption that each cell was formed at the starting point of a dislocation line, we obtain a dislocation density of 10^8 to $1.3 \times 10^9/\text{cm.}^2$ which agrees in order of magnitude with measurements of aluminium crystals.

The origin and development of the cell structures in pure aluminium after the action of the Alcoa R-5 polishing bath must well take place as follows: the attack begins locally at points with lattice dislocations which may possibly contain traces of foreign metal (Fe, Cu) as Cottrell clouds. Individual, isolated, flat etch pits (fig. 12) arise, which increase in size after further penetration of the etchant, and finally collide, thus producing the raised, rib-like cell boundary. The deposition of corrosion products on the cell ribs which is often observed (figs. 9 and 11) permits the assumption that these deposits represent the anodic part of the local galvanic cell, whereas the cell centre is cathodic in relation to the cell ribs.

From the evidence that the cells have had their origin in the etch pits, we also have an explanation for the relationship between the cell structure and the crystallographic orientation.

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BOOKS

Zinc, the science and technology of the metal, its alloys and compounds

American Chemical Society Monograph No. 142. Edited by C. H. Mathewson. Reinhold Publishing Corporation, 1959. £7 16s.

THIS MONOGRAPH is one of the very valuable series published by the American Chemical Society. Though called a chemical monograph, it is written almost entirely from a metallurgical point of view and can be thoroughly recommended as a very comprehensive and useful reference book.

It is very well printed on good paper and runs to over 700 pages. The diagrams are usually clear and helpful, but the photographs add little information and could well have been left out. For instance, a photograph of a fork-lift truck carrying bundles of zinc hardly justifies the cost of its block.

The book is written by 52 contributors, most of whom are industrial metallurgists. All aspects of zinc from its historical background to its biological significance are discussed. However, over 600 pages of the book are on the metallurgy of zinc, of which about half are on its extraction and refining.

Though the major use of the book will be as a reference, it is very readable, especially the descriptions of extraction processes. Students will find their library copy very useful (they will hardly be able to afford one of their own), though they may find the book as a whole rather thin on theory; it is in fact mainly a descriptive work.

Filler metals for joining

By Orville T. Barnett. Reinhold Publishing Corporation, New York, 1959.

THIS BOOK sets out to be a very complete guide to the filler rods, e.g. welding rods, brazing rods, solders, etc., available in the United States. They are listed under their American welding society—American Society for the Testing of Materials Classification Numbers. Under each electrode is given a description of its type-coating use and reasons for its popularity or lack of popularity. It appears to be authoritative and complete and should be extremely useful to welding engineers in America or those using American welding rods. Its use in the United Kingdom is likely to be limited.

J. H. RENDALL

Hydraulic oils

Published by Wakefield-Dick Industrial Oils Ltd., 1960. Pp. 74.

THIS BOOK, which contains 50 illustrations, will be useful to engineers concerned with the design, installation, operation or maintenance of hydraulic equipment. It includes chapters on fundamental hydraulic principles; hydraulic systems, pumps and components; and hydraulic fluids.

Copies of 'Hydraulic oils' are available to readers of this journal, free on request to Wakefield-Dick Industrial Oils Ltd., Castrol House, Marylebone Road, London, N.W.1.

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The solidification of alloys

A. KOHN and J. PHILIBERT

At the last autumn meeting of the Société Française de Métallurgie, the authors presented a contribution of the solidification of alloys subsequently published in the 'Revue de Métallurgie,' April, 1960. The following article, which will be concluded next month, has been specially prepared by the authors to give the main conclusions of their research without entering into the detailed experimental evidence presented in their original study. The authors are with the Institut de Recherches de la Sidérurgie, France, Mr. Kohn being head of the Special Metallography Division

THE MINOR HETEROGENEITY which is always found in alloys is the direct result of the process of solidification of the metal. It is now more than 50 years ago that Portevin,¹ by applying to metallic solutions the thermodynamic criteria of Gibbs, explained how liquation caused heterogeneity of the crystals formed during solidification of an alloy. The work done during the last 10 years, notably in the field of the structure of metals in the liquid state, and of phase transformation phenomena, enables more precise explanation to be made of this interpretation, which has for a long time been the standard one, by defining the nature of the processes which take place during solidification.

Crystal nucleation

When the mechanism of solidification is being considered it must not be forgotten that this phenomenon, like most phase transformations, takes place in two stages; crystal nucleation and then growth of the crystals. The second of these processes is exothermic and consequently occurs spontaneously as soon as the metal can give up calories to the outside surroundings. On the contrary, nucleation requires that a certain amount of energy shall be freely available inside the liquid phase and it can only take place if this phase is in a state of thermodynamic disequilibrium. To put it another way, a pure metal heated above its melting point can only exist in the liquid state but, below that temperature, it can exist, either in the solid state (stable condition) or in the liquid state (metastable condition). Since the work done by Turnbull, various investigators have been able to observe that a number of metals, including iron, can remain liquid at temperatures several hundreds of degrees below their melting point.²

Actually all industrial metals and alloys contain impurities which help the nucleation of crystals (heterogeneous nucleation) and undercooling rarely exceeds a few degrees. But, however small may be this undercooling, it necessarily occurs, even in those metals and alloys where there has been no opportunity of observing it, since only a definite thermodynamic disequilibrium can cause the change from the liquid to the solid state.

Therefore, the first solid nuclei are formed at a temperature lower than the melting point. The heat then freed by the growth of these nuclei raises the temperature of the metal up to its melting point (liquidus temperature in the case of an alloy).

The nature of the foreign embryos acting as catalysts in the solidification of the metal is still absolutely unknown. It can only be surmised that long distance liaison forces, similar to those maintaining the organization of atoms within a metal crystal in process of formation, act on the atoms of the liquid metal phase in the vicinity of these embryos. In other words, as various authors have suggested,^{3, 4} these embryos have a crystalline structure and crystallographic orientation exists between these embryos and the originated metal crystals as in epitaxy. It can equally be considered that the number of crystals which are formed inside a metallic mass of commercial purity is determined by the number of embryos capable of being active, which exist in the liquid phase at the moment of cooling. (This idea is implicitly the basis for all the inoculation processes destined to refine the grain of an alloy casting.)

Crystal growth

Once nucleation has started the solidification proceeds by the growth of these nuclei. It takes

place at a constant temperature in the case of a pure metal, and over a certain range of temperature in the case of an alloy. In the latter case, the chemical composition of the liquid and solid phases evolves in accordance with the progress of solidification. The crystals formed are, in general, less rich in the alloying element than was the original liquid and the latter is consequently enriched in this element.

It is usually assumed that the rate of diffusion in the solid state is very low compared with the speed of crystal growth, even in the case of slow solidification, and the homogenisation of the crystals, which can result from diffusion, is practically negligible. It is also considered that the ratio between the concentrations of liquid and solid phases, on each side of the surface of a growing crystal, corresponds to the ratio existing when the phases are in equilibrium. There is at the present time no grounds to reject this hypothesis.

But it is generally admitted that the liquidus phase can be regarded as homogeneous. Now, present data of the coefficients of diffusion in the liquid state around melting point, show that these coefficients are too low (about 10^{-5} cm.²/sec.) for an appreciable homogenization of the liquid phase to be produced by diffusion. Moreover, even if in the liquid metal there are convection currents as strong as those which appear to have been found in recent experiments, because of their laminar nature, these convection currents would not exert any influence on the liquid in the immediate neighbourhood of the crystal faces (the speed of displacement of a laminar liquid is nil when it comes up against a wall). The result of this is that the excess alloying element rejected by the crystals during their growth has no possibility of being spread out homogeneously in the portion of the alloy which is still liquid. It is for this reason, as Rutter and Chalmers⁵ have pointed out as early as 1953, that the concentration of the alloying element in the liquid near the interface must be much higher than the initial concentration found in the liquid at some distance from that interface.

Purpose of investigation

In this investigation the main interest has been given to the case in which solidification takes place with a relatively slow speed of cooling and where there is not a very high temperature gradient inside the mass of metal not yet completely solidified. These thermal conditions, which cause the formation of equiaxed crystallization, are those most frequently found in industrial practice, since they appear during the solidification of sand castings and in the core of forging ingots.

Empirical examination of a metal which solidifies under these conditions, shows that solidification first appears as a transient condition between the

initial liquid state and the final solid state, generally known as 'pasty state.' As is known, this appearance is due to the fact that solidification takes place by the growth of separate crystals, which develop at the expense of the portion of the metal which is still liquid. This mixture has neither the fluidity characteristic of the liquid state nor the cohesion characteristic of the solid state.

Although the original aim of the authors was, consequently, to go more deeply into the mechanism of solidification in ingots of killed steel, reasons of experimental convenience determined the greater part of the work being carried out on alloys with low melting points. The similarity of some phenomena observed during this investigation when compared with results obtained on steel ingots, permits a generalized interpretation to be drawn from these tests.

Thermal analysis

TEST CONDITIONS

In order to work under experimental conditions not too far removed from the usual conditions of solidification of industrial alloys, in which the presence of numerous impurities helps nucleation, raw materials of suitable purity have been used without making any effort to get an extreme degree of purity (second melt aluminium, 99.98% pure, electrolytic copper, tin, lead, bismuth). Temperatures were measured by means of chromel-alumel thermo-electric couples, 0.32 mm. dia. (0.013 in.).

With the exception of some control tests, the couple was used without protective sheath and the weld was in direct contact with the alloy, which allowed it to get into temperature equilibrium almost instantaneously with the surrounding metal. The couples were connected up to M.E.C.I. recording potentiometers, which made a continuous recording of the curves of variation of the e.m.f. of the couples as a function of time.

Tests were carried out on small amounts of metal, melted in alumina crucibles (about 1 c.c. and 16 c.c. capacity) or in nickel crucibles (about 24 c.c.), cooling being done either in still air or in the furnace, by switching off its current or by reducing the supply voltage. The device in use also allowed of rapid quenching of the crucible into a container filled with water at a given moment of cooling.

By these means cooling speeds (measured on the portion of the curve corresponding to the cooling of the liquid before solidification) could be obtained ranging from 180°C./min. to 50°C./min.

Tests were also made on larger amounts of metal (1,800 g. (4 lb.)) approx., by using a cylindrical alumina crucible 120 mm. high inside and 95 mm. inside dia., placed on a metal base to which were fixed the thermo-electric couples penetrating into the metal along the axis of the crucible. This

device could be placed in a furnace where the heating was done, then withdrawn and placed on a frame so as to obtain air cooling (speed of cooling about 15°C./min.). Tests were also made by leaving the device in the furnace, with the current cut off (speed of cooling 2°C./min.).

Solidification curve of pure metal. The curves obtained by leaving to cool, in still air or in the furnace, a small quantity of tin (150 g., 5½ oz.) or of aluminium (3-40 g.), containing no alloying element, consist of the three usual sections (fig. 1): (1) The cooling curve in the liquid state; (2) the plateau of solidification; and (3) the cooling curve in the solid state.

The first two sections fit together with a dip corresponding to supercooling. This latter is higher and stops more quickly with tin than with aluminium.

Various tests have also been made with the large crucible containing 1.8 kg. (4 lb.) of aluminium, with the joints of the three couples placed respectively in different positions on the vertical axis of the crucible. (Fig. 2.) The recorded curves show that local re-heating (supercooling) occurred at any point of the liquid aluminium as soon as the temperature at that point reached a certain degree of supercooling. The identical shape of the supercooling dips observed on the graphs relating to any one test showed that the kinetics of this re-heating is the same at a point situated near the wall of the crucible or near the centre of the ingot. This result shows that this re-heating is due to the growth of crystals close to the couple joint. As a matter of fact, if the re-heating observed at the centre of the metal was due solely to transmission by conductivity of the heat arising from solidification of metal in

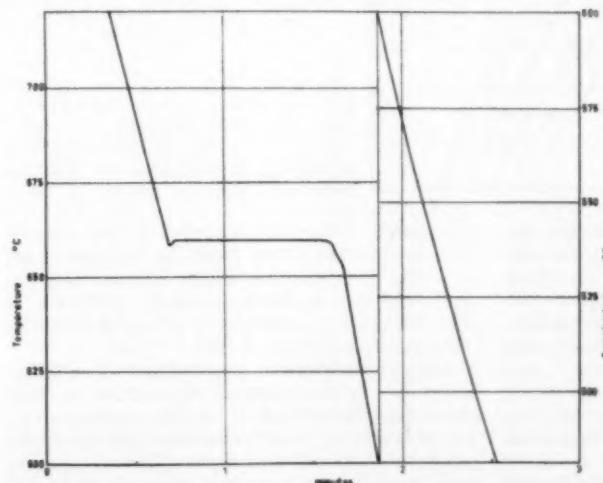
the neighbourhood of the wall of the crucible, the rise in temperature at this point would be much slower than that which would be observed near to the wall. Moreover, the thermal analysis curve would show an anomaly indicating the moment at which nucleation took place at that point in the ingot, which is not the case. It should be noted, however, that solidification near the wall is only completed after a period of time corresponding to two-thirds of the total length of time of solidification.

These observations show, then, that contrary to a widely held opinion,⁶ solidification of a non-alloyed metal is not necessarily carried out by the progressive thickening of a skin of solid metal enclosing a volume of metal still completely liquid. This phenomenon can only happen in this way when the thermal conditions cause the formation of basaltic crystals growing simultaneously from the surface of the ingot.

Influence of increasing amounts of added alloying element. In order to know the influence of an increasing addition of alloying element on the shape of the solidification curves, tests have been carried out on tin-bismuth alloys (up to 3.1% Bi), tin-lead (up to 2.7% Pb), and aluminium-copper (up to 3.6% Cu) by making successive additions of the alloying element to the same base metal.

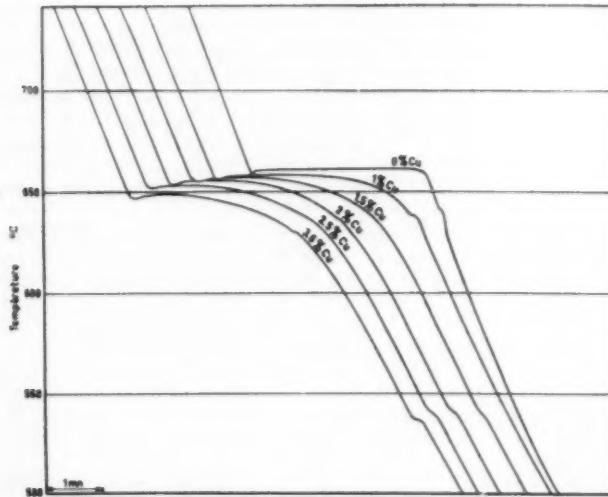
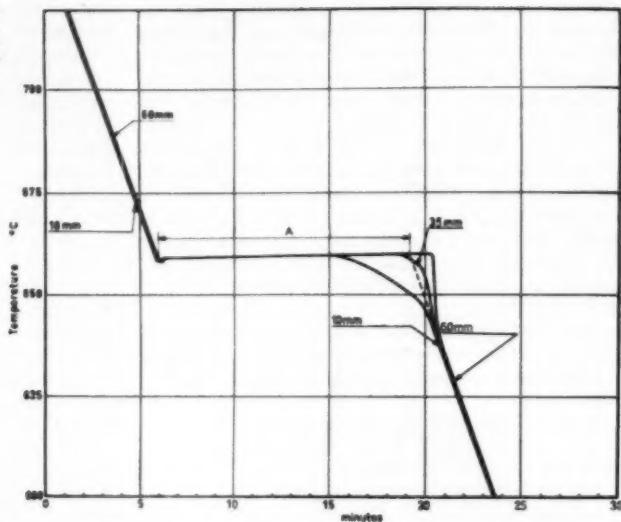
The solidification curves of the three series of alloys shows the same appearance as those shown on fig. 3, reproducing some of the curves obtained with aluminium-copper alloys:

- (1) Solidification always takes place after a very obvious supercooling.
- (2) The addition of a small amount of alloying element results in an increase in the radius of curvature of the concave portion shown at



1 Thermal analysis curve of a 3 g. sample of pure aluminium cooled in air in an alumina crucible. (Rate of cooling: 180°C./min.)

2 Thermal analysis curves recorded by 3 couples placed at 10, 35 and 60 mm. from the base of a refractory crucible containing 1,800 g. of pure aluminium. (Rate of cooling: 13°C./min.)



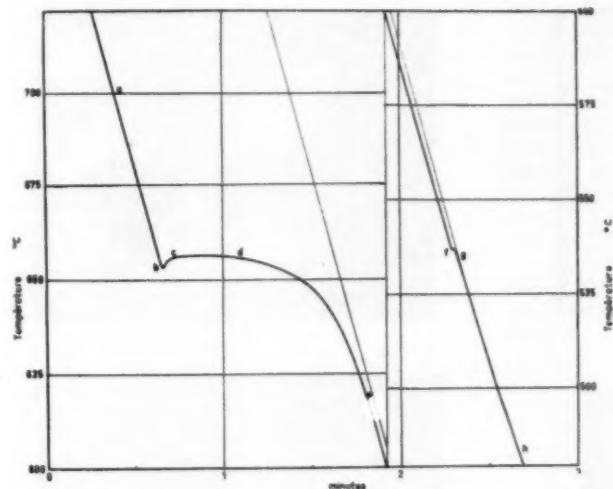
3 Thermal analysis curves of aluminium alloys with increasing amounts of copper

the end of the plateau. The higher the amount of added element, the greater the radius of curvature. But, even in tin alloys with 2.7% Pb, or 3.1% Bi, and in the aluminium alloy with 3.6% Cu, the solidification curve shows a portion practically horizontal after the supercooling dip.

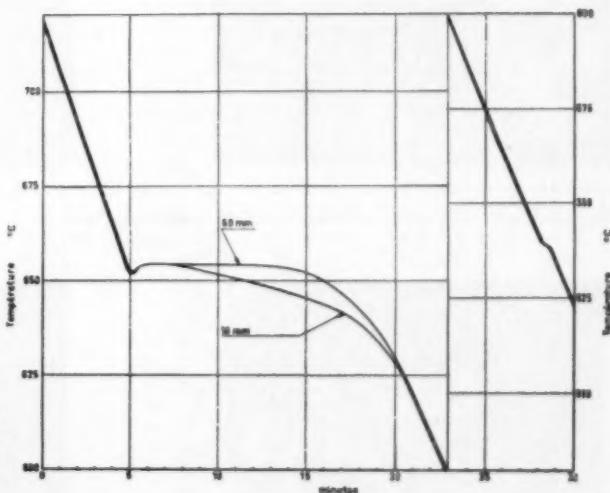
- (3) The slope of the straight-line segment which follows this concave portion is practically the same for a pure metal and for its various alloys.
- (4) For most of the alloys investigated, the

graph shows an anomaly at the eutectic temperature, even when the amount of the added element is much smaller than its solubility in the basic metal: 1.5% Cu in aluminium (solubility 5.65%); 0.40% Pb in tin (solubility 2.5%).

Study of solidification of aluminium-copper alloys (2% Cu). It was required to ascertain to what extent the general shape of the alloy cooling curves was influenced by the experimental conditions under which solidification was done. For this reason, more particular study has been made of alloys with



4 Thermal analysis curve of a sample of 3 g. of Al-Cu alloy, with 2% Cu, cooled in air in alumina crucible. (Rate of cooling: 180 C./min.)



5 Thermal analysis curves recorded by two couples placed at 10 and 60 mm. from the bottom of a refractory crucible containing 1,800 g. of Al-Cu alloy, with 2% Cu. (Rate of cooling: 13°C./min.)

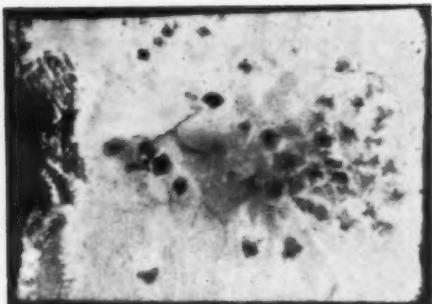
about 2% copper, this copper content having been chosen so as to allow of distinguishing definitely when solidification ended by means of the special characteristic of eutectic deposit.

These tests were carried out with changes made in the amount of alloy used (3, 60, 1,800 g.) and also in the cooling rates, that is, in air (fig. 4) or in the furnace. When tests were made with weights of 1,800 g., cooling curves corresponding to two or three different points of the small ingot were simultaneously recorded. (Fig. 5.)

Study of numerous curves obtained during these tests shows that the solidification of a 2% Cu-Al

alloy takes place under the following conditions, which can be seen, for example, on fig. 4:

(1) The beginning of solidification is shown by a slight rise in temperature (section bc), after a supercooling of 1°C. to 3.5°C. (It can be noted that a reheating of 1.7°C. of some aluminium corresponds to the heat given off by the solidification of 0.5% of this same amount.) This phenomenon can be considered as practically simultaneous in the whole of a volume of the order of a dm.³ ignoring the slight gradient in temperature present during cooling in the liquid state. It seems that the smaller the mass of cooling metal, or the



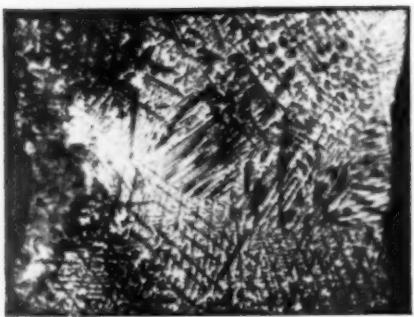
(a)



(b)



(c)



(d)

6 Autoradiographs taken of samples cooled slowly and then quenched at various intervals of time after nucleation. (a) Quenched after 25 sec., (b) after 5.5 min., (c) after 10 min., (d) completely cooled in furnace



7 Comparison of the macrographic structure (left) and the autoradiograph (right) of a specimen cooled at 6°C./min. Then quenched during solidification $\times 15$

higher rate of cooling, the greater the degree of supercooling.

(2) During the first part of solidification, a large fraction of the alloy solidifies at a temperature which varies little (section CD) and the heat given up to the outside surroundings is due for the major part to the latent heat of solidification produced.

(3) After this first period there is a transitional period (section de) during which the kinetics of solidification and the change of temperature at a point in the alloy depends on the speed of release of calories through the outside surroundings and on the total mass of the alloy.

(4) During the third period of solidification, which begins when the largest part of the alloy is already solidified, the residual liquid solidifies over a large range of temperature (section ef). The heat released to the outside surroundings is almost entirely caused by cooling of the alloy.

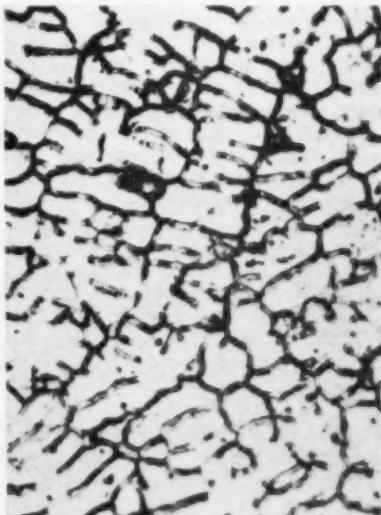
(5) Solidification is completed by a small deposit of eutectic (anomaly fg) which takes place practically at the same moment in the whole of a volume of the order of 1 dm.³ Analysis of the curves, obtained during cooling in air or in furnace, of small quantities of alloy, shows that the proportion of eutectic formed is of the order of 3% in this case. This anomaly is generally noticed at a temperature below that of the eutectic point, the margin reaching as much as some 10° in the case of rapid cooling.

Autoradiographic examination

This has been carried out on samples of aluminium alloy containing about 2% Cu, prepared with the help of the apparatus used for thermal analysis. About 50 g. of alloy were melted in a small nickel crucible and the unprotected joint of a chromel-alumel couple was put into the inside of the liquid metal. The crucible was allowed to cool inside the furnace (6°C./min.) or in air (45°C./min.), whilst a recording was made of the cooling temperature as indicated by the couple. At a certain stage of solidification the alloy was drastically cooled by plunging the crucible into salt water to within $\frac{1}{4}$ in. of its top edge. Under these conditions it was possible to get an extremely swift solidification of the metal which was still liquid at the moment of quenching.

From the alloys, thus solidified, thin sections were cut and these were irradiated so as to activate the copper, producing radiocopper 64 with a half-life of 12.8 h. These samples have enabled autoradiographs to be made showing the distribution of copper, as given on figs. 6 and 7.

Microscopic examination, with a small magnification, of specimens electrolytically polished and lightly etched with Keller reagent, has shown that the crystals formed during cooling in the furnace were large dendrites visible to the naked eye, the branches of which were sufficiently spaced to be distinguishable on the autoradiograph. The liquid metal still at the moment of quenching was composed of small dendrites, the details of which were



8 Structure of Al-Cu (2% Cu) specimens etched with 3-acid reagent. (Left) Specimen cooled in air at 180°C./min. (Right) Cooled at 6°C./min., then quenched during solidification. $\times 50$

below the power of resolution of the autoradiograph. (Fig. 8 (a)). On this occasion we have been able to confirm the observation made by Alexander and Rhines,⁷ according to which the ramification of the dendrites is by so much more intense (and, consequently, the distance between the branches of one same dendrite is by that much smaller) as the speed of solidification is so much higher. (Fig. 8 (b).) The average width of a dendrite branch, measured on specimens cooled at very different speeds, is given in the table.

Rate of cooling in liquid state	Time for solidification (between supercooling and eutectic deposit)	Mean width between branches (μ)
Very quick (in water)	A few seconds	10 to 20
180°C./min.	1.65 min.	30 to 40
6.5°C./"	23 "	200
In furnace	250 "	1,000

Examination of the autoradiographs leads to the following conclusions: (1) At the moment when supercooling stops, small crystals are present inside the liquid metal. They originate in any points whatever, without being helped to nucleate by the presence of a nickel crucible (or an aluminium or porcelain one, as used in preliminary tests), of the couple or of its protective sheath. This result confirms and completes observations previously made on iron-sulphur and iron-phosphorus alloys.⁸

(2) The crystals thus formed have a copper content much lower than that of the metal still liquid at the moment of quenching.

(3) The autoradiographic image of the samples quenched when the temperature indicated by the couple had dropped 2.5°C. below the maximum temperature recorded at the beginning of solidification, is similar to that of the check-samples which were cooled in the normal way (micrographic examination, however, shows in the quenched specimen very ramified fine dendrites, which are not visible in the specimen completely cooled in the furnace). These observations confirm that, at the moment when the samples were quenched, the core of dendrites was completely formed and that the only metal which remained liquid was in the inter-dendritic spaces highly enriched with copper (about 30% of the total volume of the sample).

Analysis by Castaing microprobe

ALUMINIUM-COPPER ALLOYS

Analyses with the Castaing microprobe have been made at different points of six samples of aluminium-copper alloys solidified under different conditions: two of these specimens, used for autoradiographic study, have been quenched during the

process of solidification. These analyses have been carried out in two different ways:

(1) *In the usual way with a beam of electrons suitably focused (dia. about 1 μ).* In this way determinations have been made of very localized variations of copper concentration inside a dendrite branch entirely formed during continuous cooling, slow or quick, or inside a dendrite branch the growth of which had been interrupted by very rapid chilling of the specimen.

(2) *By defocusing the electron beam so as to be able to determine the average copper content of a volume with a thickness of about 1 μ (penetrative power of electrons into specimen) and a circular area of 50 to 60 μ dia.* In this way it has been possible to study, in the samples quenched during solidification, the average chemical composition of the metal which was still liquid at the moment of quenching, in spite of the heterogeneity set up by solidification, formation of closely branched dendrites.

to be continued

Relationship between core and case

Nearly ten years of research, which indicates that the hardenability of both case and core of carburized steels is essential for the proper selection of the optimum grade and control of its processing for a specific part, is summarized in a book, 'ATLAS, hardenability of carburized steels,' published by Climax Molybdenum Company, a division of American Metal Climax, Inc.

For many years it was believed that core hardenability alone was needed to ensure adequate case hardenability. Research, however, has disproved this concept with evidence that equal additions of carbon do not have the same effect on the hardenability of base compositions. This is documented in the new book with more than 125 charts which show the effect of cooling rates and distance from end of specimens on the Rockwell Hardness of a number of carbon, molybdenum, nickel-chromium, nickel-chromium-molybdenum and other steels.

Blanket for 'Bluebird'

One of the problems confronting the designers of Donald Campbell's new *Bluebird* was that of protecting the structure from the heat of the gas jet, the temperature of which is about 500°C. This is only a few degrees below the melting point of the light alloy of which the car is made. An efficient and light-weight insulation to fit round the jet pipes was provided by 'Refrasil,' a material that has been used increasingly over the last five years in most British and European jet aircraft.

'Refrasil,' made by the Chemical and Insulating Company and marketed by the British Refrasil Company, both members of the Darlington Group, is a virtually pure silica fibre which looks like glass fibre and comes in the same form. It has a melting point half as high again as that of mild steel with a very low thermal conductivity.

On *Bluebird* the saving of weight is imperative and as a fixed insulation was possible, an integral blanket is fitted in which the batt, or felt, is enclosed between an outer skin and the component itself, which acts as an inner skin. The skin is made of 0.007 in. plain and dimpled stainless steel and the insulation filling is three layers of batt interleaved with Alfol in sheet form. This fits closely round the four-branched exhaust, which passes above and below the rear wheel driving shafts.

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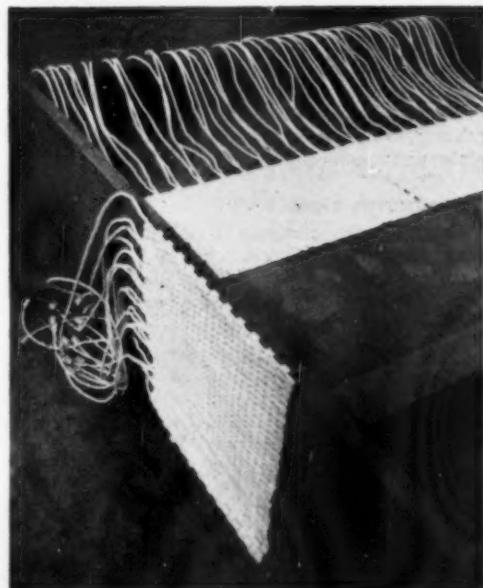
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Electrothermal stress relieving

Treatment of 80 ft. fabricated bridge girders

STRESS RELIEVING of very large or long welded fabrications can present quite a problem if suitable furnace accommodation is not available. This difficulty arose recently after the fabrication of four huge steel girders at the Chepstow works of the Fairfield Shipbuilding and Engineering Co. Ltd. for the new Kuncorn-Widnes road bridge, which will ultimately replace the existing transporter bridge.

Some idea of the size and design of these girders may be gathered from the illustrations. Each completed girder was approximately 80 ft. long by 6 ft. high and weighed in the region of 14 tons: they were welded fabrications constructed with $\frac{3}{8}$ -in. thick webs and $1\frac{1}{2}$ -in. thick flanges, and stiffened at regular intervals with 8- by $3\frac{1}{2}$ - by $\frac{1}{2}$ -in. angles. For convenience of manufacture each girder was fabricated in two separate 40-ft. lengths, which were then joined by welding to produce the complete 80-ft. girder.



As these girders will carry most of the load, very strict specifications were laid down regarding the stress relief procedure for the weld joining the halves of the girders. These were as follows: (1) heat to 650°C . at a speed not exceeding 150°C./h. , (2) soak for a period equal to 1 h. in. of the thickest part of the heated area, (3) cool to 350°C. at a rate not exceeding 100°C./h. and (4) allow to cool naturally to room temperature.

Flexible furnaces

No conventional furnaces capable of accommodating such large girders were available at the Chepstow works and Electrothermal Engineering Ltd. were invited to undertake the stress-relieving operation, using their 'Flexible Furnaces.' These are, in effect, mats composed of ceramic beads threaded on 'hairpins' of nickel alloy resistance wire: the latter are linked together to provide a series of electrical circuits capable of giving a maximum mat surface temperature of $1,000^{\circ}\text{C.}$ Each circuit is provided with flexible leads, and by bringing in or cutting out circuits it is possible to

1 LEFT 'Flexible Furnace' in position on girder

2 BELOW The prime lagging being applied to upper face of girder



control the mat temperature to close limits. The mats are very flexible, and in one direction can be bent to a minimum radius of 1 in.; thus they may be wrapped around the edges of plate, etc., without difficulty (fig. 1).

Problems involved

Before commencing the treatment it was necessary to give careful consideration to certain problems arising from the close temperature cycle specifications, the considerable mass of metal involved and, in particular, the large surface area from which heat could be readily dissipated. In particular, it was necessary to determine (1) the amount of heat input required, (2) the power necessary to provide this heat and (3) the amount of lagging necessary to conserve the heat input. Also, because of the weight and size of the girders, the services of two cranes were required each time they were moved: consequently, forward planning of the movement of girders was essential because the cranes were also employed on normal shop work and were not always readily available.

Heat input. With regard to the question of heat input, while it was known that the flexible furnaces were capable of producing a maximum temperature of 1,000°C. and could dissipate 50 W./sq. in. it was appreciated that they would have to be considerably underrun in order to keep to the specifications. In fact, the density actually employed was well under 25 W./sq. in.

Power supply. The power for heating purposes was supplied by two standard 300-amp. quasi-arc and two English Electric 350-amp. welding transformers; these are continuous rating amperage figures. By means of a standard earth clamp the end of each transformer lead was connected to a copper busbar, to which were attached leads from several mats. As the attachment was made by means of nuts and bolts, it was an easy matter to disconnect or connect leads in order to lower or increase the temperature in any particular area as the need arose.

Thermal insulation. Four-inch thick slab-type thermal insulation material was employed for lagging purposes. Two types were used, the prime lagging (adjacent to the metal surface) being capable of efficient results at temperatures up to approximately 800°C.; the outer or secondary lagging was also extremely efficient, but had an upper limiting temperature of about 500 to 600°C.

Before commencing the lagging operation it was necessary to decide the size of the area to be lagged, the number and position of the heating mats, and the number and position of the thermocouples to be used for temperature control purposes. These details were then marked on the girder.

It was decided that a minimum distance of 3 ft.

on each side of the weld should be lagged, and prime lagging was applied to the underside of the girder and to the inside of the flanges (on top of the mats and thermocouples), and secondary lagging then added with the girder in the vertical position, the lagging being held in position by means of boards and wire rope. The girder was then turned through 90 deg. to the position seen in fig. 2, and the upper surface lagged in a similar manner, the total thickness of lagging being in the region of 16 in.

Temperature recording. The flexible furnaces were positioned under the prime lagging, and at strategic positions under the mats were thermocouples connected to Type 800 ether temperature recorders. Special precautions were taken to ensure the uniformity of all the thermocouples: for this reason they were immersed in boiling water and the recorders checked to ensure that each gave a reading of exactly 100°C. This was considered a sufficiently good check that they were all accurately matched. It was, of course, also essential to ensure that all the leads were exactly the same length, in this case 15 ft.

It should be noted that the thermocouples not only provided a means of recording the temperature in the areas concerned, but were also used to monitor the mats or sections of mats. In other words, the operator watching the recorders could predetermine whether the temperature in any particular area under treatment should be increased or decreased. This was achieved by carefully studying the general trend of the curves, and from this information determining what would happen in the near future.

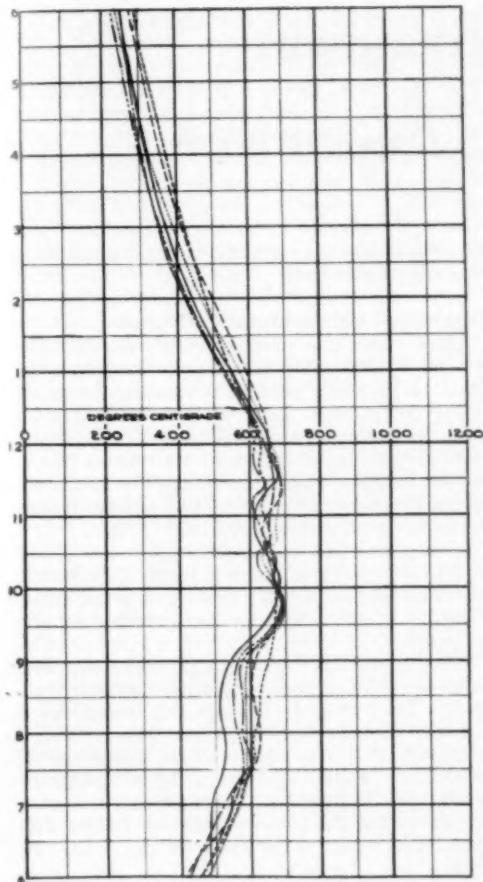
In all, 17 thermocouples were positioned at various points around each girder, some in contact with the metal surface and others buried in the lagging. By this means very useful information was obtained relating to the temperature gradients along the girder and also through the lagging.

Temperature control

Control of the heat input in order to comply with the specification cycle was achieved by varying the amperage output of each transformer, and at the switch-on stage the tappings were the lowest possible obtainable.

The voltage and amperage readings were made in the conventional manner, using voltmeters and tong testers. Bearing in mind that welding sets by different manufacturers and of varying age have different drooping characteristics, all measurements were taken at the terminals of the flexible furnaces.

Any tendency for one area to heat more rapidly than another was corrected without difficulty merely by cutting out of circuit the appropriate mats or sections of mats, and thus it was comparatively easy to ensure uniform heating over the entire weld area.



3 Typical temperature record during a heat-treatment cycle. Controlling individual mats enables all six readings to be kept uniform

The general rise in temperature during heating was maintained closely to specification requirements by stepping up the output of the power sources to suit the readings shown on the recorders, at the same time adjusting any local difference in the manner just described. When the temperature in all areas reached approximately 575°C ., i.e. about 75° below soaking temperature, the voltage and amperage output of the sets were reduced in order to avoid any chance of over-shooting the maximum temperature. It was found that interpretation of the curves shown on the recorders enabled the operators to predetermine very accurately the change of settings required.

Soaking was achieved comparatively easily, and the subsequent cooling-down stage amounted to little more than intelligent interpretation of the recordings. During the stage of cooling from 650 to 350°C . the furnaces were switched on and off as necessary to maintain a steady rate of cooling in each area; this uniformity of cooling was made possible by the fact that each heater could be independently controlled. When the temperature had fallen to 350°C . all heaters were switched off and the entire assembly allowed to cool naturally without disturbing the lagging.

After the cycle had been in operation for some time, heat rising from the lower portion of the vertical flanges increased the rate of heating of the upper portion. For this reason it was essential to be able to control the heating of these upper surfaces by putting the appropriate mats out of circuit until the temperature was uniform throughout the entire flange, as shown by the thermocouple readings.

The very accurate control of temperature achieved at all stages was undoubtedly influenced considerably by the careful attention paid to the lagging operations. Heat losses were minimized, thereby conserving energy input and, more important, temperature gradients through the girders were comparatively small. In view of the size of the girders, the mass involved and the considerable surface area, problems could readily have arisen if the lagging had been skimped or done carelessly, and a good deal of the success of the heat treatment of these girders was definitely attributable to the amount of time and forward planning given to the question of thermal insulation.

Typical treatment cycle

Fig. 3 is an actual temperature recording made during a heat-treatment cycle. It will be seen that at the commencement of the cycle the rate of temperature increase was slower than that called for by the specification (150°C .). This was done deliberately, since, on switching on, a number of things happen before the heat is being actually transferred to the girders in a manner which will give a good heat treatment throughout the cycle. The slowness of heat input resulting from passing only a very small amperage through the mats provided a fair amount of time for the thermal inertia of the flexible furnace, the lagging and girder to be overcome, and reduced to an absolute minimum the possibility of major temperature gradients from one face of the girder to another, or from one side of the flanges to the other.

After running the heaters up to about 200°C ., the rate of temperature rise becomes rather more rapid, but still within specification limits. Up to 650°C . it will be seen that, by and large, all the curves are well within the specified $150^{\circ}\text{C}/\text{h}$.

Fully automatic heat treatment

Installation at British Timken's Daventry Works

BRITAIN'S FIRST completely automatic heat-treatment line for carburizing, annealing, hardening and tempering has been installed by AEI-Birlec Ltd. at British Timken's Daventry East Works. The equipment has been built primarily for the heat treatment of tapered roller bearing cups and cones for vacuum brake rolling stock required under the railway modernization scheme.

The processes, which are in automatic sequence, are performed in four Birlec furnaces. The furnaces are linked by mechanical and hydraulic handling devices designed by British Timken to meet their specific requirements. The installation performs the following processes: (1) Gas carburizing; (2) sub-critical annealing; (3) hardening; and (4) tempering.

Prior to heat treatment, cups and cones are stored in multiple units. From there they pass through a washing machine to the furnace operator's loading station. The components are loaded by hand on to locating fixtures manufactured from high-grade cast nickel-chromium alloy. The loaded fixtures are assembled four at a time on nickel-chromium trays, three of which form a furnace charge at every loading sequence. Once the components are loaded on to the trays, their progress through the complete heat-treatment cycle is fully automatic. No further manual operations are required until

the components are unloaded on emergence from the tempering furnace.

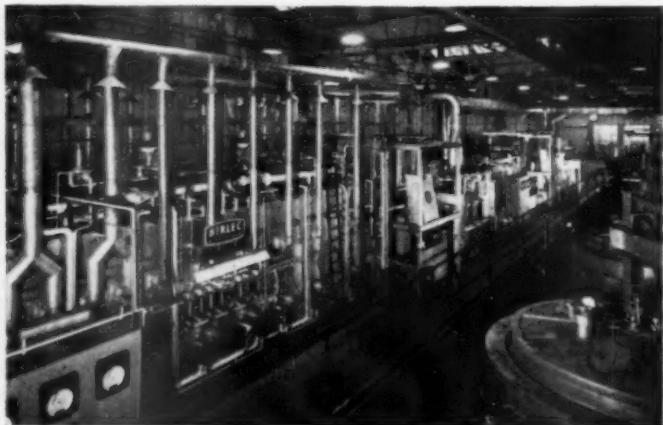
Continuous gas carburizing furnace

Three loaded trays simultaneously enter the vestibule of a continuous carburizing furnace. The furnace is heated by gas-fired radiant tubes arranged in four zones, each under independent and automatic temperature control. The first of these is a heating zone, the middle two are soaking zones, and the fourth is a diffusion zone. The total cycle time in the heating chamber is 24 h., and the furnace has a capacity of approximately 500 lb. h.

A controlled carburizing atmosphere is supplied to the furnace by an adjacent Birlec endothermic generating plant. Propane enrichment of the atmosphere is regulated by a Foxboro-Yoxall dewpoint controller.

The trayloads of bearing cups and cones that comprise a furnace charge are aligned along a triple track. They progress through the furnace by a sequence of synchronized pushes from hydraulic cylinder mechanisms located at the charge end of the furnace. Whilst, at present, identical time cycles operate on the three tracks, appropriate sequence equipment has been incorporated to enable different time cycles to be used on the individual tracks.

*Birlec gas-fired triple-track pusher-type furnace rated at 3,500 cu. ft. h. and leading into a single track with intermittent pusher furnace.
Installed at the Daventry Works of British Timken, Division of the Timken Roller Bearing Co., it is part of the continuous line for tempering, annealing and hardening bearings*



The heating chamber is 31 ft. 6 in. in length and has a cross-section measuring 6 ft. 4 in. wide by 1 ft. 6 in. high. Safety features have been provided so that, in the event of a power failure, the outer furnace doors rise automatically and the furnace is shut down in a 'fail-safe' manner.

At the discharge end of the furnace a further hydraulic pusher moves one tray at a time in sequence from the three tracks on to a platform. The platform is then lowered automatically into an oil tank to quench the charge. Next, the loaded tray is transferred by a motor-driven chain conveyor through a washing station. After passing through the two compartments of the station, one for spray wash and the other for a hot-water rinse, the work tray is transferred automatically to the loading mechanism of the sub-critical annealing furnace.

Sub-critical annealing furnace

The second furnace in the installation, for sub-critical annealing, is an electrically heated pusher-type furnace. Rated at 145 kW., it is heated by wall-mounted tape elements arranged in three zones. Cups and cones are annealed in the furnace governed by the automatic cycle of the carburizing furnace. An exothermic generating plant supplies a protective atmosphere to the heating chamber. After annealing, the trays are discharged singly from the furnace and quenched, washed, rinsed and dried in a manner identical to that employed for carburizing.

At this stage in the heat-treatment cycle the components are 'de-stacked' from the tray and delivered to conveyors loading the hardening furnace. 'De-stacking' is performed by two sets of air-operated fingers projecting from an overhead

trolley. The stacks of cups and cones are lifted off the tray fixtures and released in pairs on the conveyors. In the second stage of this operation the turntable supporting the tray is rotated through 180°. The trays and fixtures thus emptied are returned to the charge end of the carburizing furnace by a chain-driven pusher and conveyor.

Hardening furnace

Bearing ring components after sub-critical annealing in the pusher furnace are transferred to a Birlec roller-hearth furnace for hardening. The conveyors feeding the hardening furnace each carry forward a component which is deposited on the furnace track by a twin lever mechanism synchronized with the opening of the charging door.

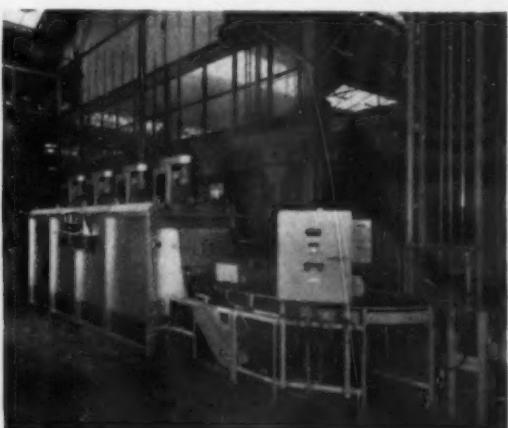
The heating chamber, which measures 18 ft. long internally, is electrically heated by nickel-chromium alloy tape elements in the roof and by cast grids in the hearth. There are two heating zones in the furnace, with a total rating of 120 kW., and each component completes the hardening cycle in approximately an hour. The furnace is provided with an atmosphere from an adjacent endothermic generating plant of a type similar to that feeding the carburizing furnace.

The rollers forming the hearth of the furnace are arranged in a number of sections. Each section is individually driven from a main shaft running longitudinally at one side of the furnace, just above floor level. At the discharge end of the furnace the last section of rollers is fitted with an over-drive for fast discharge. Two photo-cell units control the operation of the fast roller-drive gear and the discharge door. On completion of the necessary soaking time in the hardening furnace,

48 kW. Birlec slat-conveyor tempering furnace



120-kW. roller-hearth hardening furnace at Daventry



the discharge door is opened to allow a pair of components, as detected by the photo cell, to be discharged by the fast rollers. On emergence the components are turned by deflecting ploughs through 45° to a pair of jig quench machines.

Pneumatic devices position the components against adjustable stops on the quench plates of these machines. Controlled quenches are then performed by the operation of air-hydraulic rams carrying jig plugs. These locate in the bores of the components during quenching and minimize distortion during the operation. A high rate of flow of quench oil is maintained for a predetermined period. The press ram then rises and the components are discharged along chutes and conveyors to a further washing machine.

Tempering furnace

After washing, cups and cones are aligned in a mechanized handling device which feeds the components across the width of a continuous tempering furnace. This device positions the cones inside the cups before entering the furnace.

Tempering, which is the final stage of the heat-treatment operation, is carried out in a continuous Birlec slat-conveyor furnace rated at 48 kW. The furnace chamber, 15 ft. in length, is heated by special electric elements fitted in the side walls and arranged in two zones.

Components travel through the furnace on a series of steel slats supported by roller chains which engage sprockets at the ends of the frame. To promote convection heating, the slats are perforated and a vigorous air flow through the load is maintained by four roof-mounted fans. At the discharge end of this furnace, cups and cones are separated by another handling unit which so positions the components that they fall into two separate chutes. From these positions the components are fed on to two wire-mesh belt conveyors to stacker storage units identical to those proceeding the heat-treatment line. These units provide the storage of components prior to grinding.

Auxiliary services

By the correct selection of quenching operations with this continuous installation of atmosphere furnaces it has been possible to minimize the formation of scale on components during heat treatment so that there is no necessity for any form of shot-blasting operation. Furthermore, by incorporating washing, rinsing and drying operations, degreasing of components has been avoided.

The supply of quenching oil to the three quenches in this installation, as well as to the roller heat-treatment installation, is obtained from an oil service house with three 6,000-gal. capacity oil tanks. Thermostatically-controlled Heenan and Proude

New air hardening steel

A NEW STEEL, 'Airque V,' has been developed by Braeburn Alloy Steel Corp., Braeburn, Pa., U.S.A., to increase the wear resistance of the A-2 (5% chromium) air hardening steels.

Already in use for gauges, slitters and knives, dies (thread rolling, powder metal, stamping, forming and trim), and similar purposes, it has also seen service for diesel engine injection plungers. Other suggested uses include press forging die inserts, grinding machine spindles, machine ways, and swaging dies.

Air hardening

Optimum hardening temperature is 980°C., slightly higher than the AISI Grade A-2 which was its progenitor. And the alloy hardens in air, even in fairly large sections. Still air will suffice in sections up to about 5 in. thick; an air blast may be used on heavier sections (a 5-in. cube will harden in still air to about 60 Rockwell C).

Usual heating methods—atmosphere, salt bath, or pack—can be used; a 650°C. pre-heat is recommended. Tempering (double tempering is best) will require 150°C. to 650°C., depending on hardness and shock resistance required.

Distortion on hardening of the new alloy is about the same as the A-2 grades. And expansion is substantially the same across and with the grain.

Airque V has this typical % analysis:

Carbon	1.25
Manganese	0.50
Silicon	0.30
Chromium	5.25
Molybdenum	1.15
Vanadium	1.00

Principal difference between the new alloy and Braeburn's A-2 analysis (called Airque): Increased concentration of carbon and vanadium. Result of the changes: Increased hardness at all tempering temperatures (difference ranges from 1 to 3 points on the Rockwell C scale) and an increase of 11°C. in the optimum hardening temperature. And it has less tendency to retain austenite on moderate overheating.

coolers mounted on the roof maintain the correct oil temperature.

Throughout the whole of this installation and the roller heat-treatment installation the extraction of fumes and waste gases from the furnaces and washing machines has been afforded by a system of exhaust trunking supplied by the Modern Air Co., of Leicester. This trunking unites all points of fumes and gas escapement in the installation to a common junction, and thence to a fan unit mounted on the roof of the oil service house.

NEWS

Research for the engineering industry

THE NATIONAL ENGINEERING LABORATORY is giving priority to research on industrial problems of immediate importance, including investigations in the machine-tool field, the development of hydrostatic power transmissions, and work on the cold extrusion of steel.

Inaccuracies in machine tools can be greatly reduced using moiré fringe techniques. Developed jointly by the National Engineering Laboratory and the National Physical Laboratory, the system can be applied to measure the errors in the movement of the machine and to control the movements.

The error signal obtained with this technique can be fed into a servo-system and correcting mechanism to reduce errors in relative movement continuously and automatically; in an experimental rig at NEL incorporating a worm and worm-wheel drive, errors in transmission of uniform relative motion have been reduced by a factor of twenty. NEL has collaborated with David Brown Industries Ltd. in applying such an error-correcting system to the final table drive of a 30-in. gear hobbing machine. This machine and gears of much improved accuracy produced on it was on show at the International Machine Tool Exhibition at Olympia last month.

New eating places for Britain's travellers

The 'Little Chef,' a completely self-contained catering unit, has recently been introduced to the U.K. by Diners Ltd.

The unit, which is built on a heavy steel chassis, consists of a completely prefabricated permanent building of steel and aluminium, containing stainless steel kitchen catering and snack bar equipment.

Being a prefabricated building, the 'Little Chef' can be moved to a prepared site (concrete foundation with drainage and main services already provided) and in a matter of hours is off-loaded, connected up and in full operation.

All types of grills, freshly fried potatoes, toasted or plain sandwiches, hot and cold beverages, cold meats and salad dishes will be served. All food storage is refrigerated, including the sandwich display unit.

The first unit has been put into service at a site at Tilehurst, Reading, and it is hoped to develop at a present rate of two or three a week, increasing as demand requires it.

China orders British equipment

China has placed another order to the value of £8,000 for six gas-liquid chromatographic analysis apparatus manufactured by Griffin & George Ltd., Alperton, Middlesex. This is the second large order to be received from China in 12 months, making the number of these instruments now in use in China nine.

Cold extrusion of steel

The production of pins, sparking-plug bodies, ball-race housings and other hollow components by extrusion from an unheated steel billet has the advantages of a high production rate, good surface finish, and enhanced mechanical properties as a result of cold working of the metal. Machining is reduced, and may be eliminated with certain components. In many cases this would save up to 70% in material otherwise lost by machining.

Cold extrusion has been used very little in Britain, although the process has been carried out extensively in Germany and the USA. One of the reasons has been the

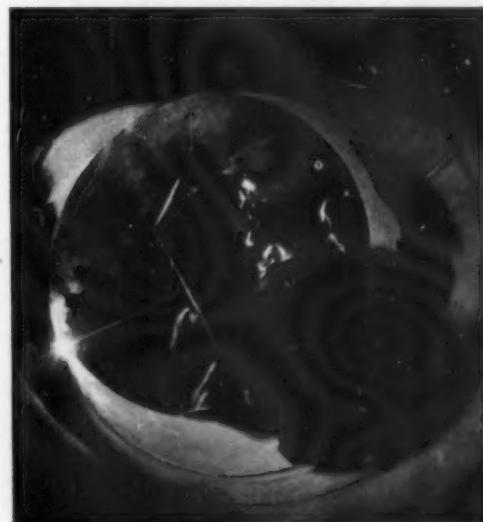
shortage of information on design procedures. Comprehensive data are now available from NEL on the effect of different process variables on the pressures required to extrude various products in a wide range of low carbon and alloy steels. Information is also available on the flow of metal during extrusion and on the properties of the extruded product. Several British firms are now trying cold extrusion of steel as a production process. The information available enables NEL to advise on the extrusion conditions for particular products and the design of appropriate tooling.

New physics laboratory at Oxford

A fundamental and rewarding field of research in physics at the present time is the behaviour of materials at very low temperatures. Investigations of this kind are an important aspect of the programme of research at the Clarendon Laboratory of Oxford University, where Professor B. Bleaney, F.R.S., is Dr. Lee's Professor of Experimental Philosophy.

A sphere of especial importance is the variation in the magnetic properties of materials at near the absolute zero of temperature and Dr. Nicholas Kurti, F.R.S., of the Clarendon Laboratory, is particularly concerned with investigations of this nature. To extend the University's efforts in this direction a new laboratory is to be built for the purpose and Mullard Ltd. have made a grant of £25,000 towards the cost of this new building. The new laboratory will be known as the Mullard Cryomagnetics Laboratory.

This section of a blast-furnace connecting main for the extension to the Irlam Steel Works of the Lancashire Steel Corporation is being fabricated at the works of Pearson & Knowles Engineering Co. Ltd., Warrington, using English Electric 'Enrox' electrodes and welding equipment. 'Enrox' is a general-purpose, mild-steel electrode suitable for welding into any position



Lectures and conferences
Conference on quality control

An international conference on the subject of quality control will take place at Church House, Westminster, on September 1, 2 and 3, when some of the world's leading authorities on quality control will participate.

The conference, annual event of the European Organization for Quality Control, will be opened by Mr. Frederick Erroll, Minister of State, Board of Trade, supported by Sir Bertram Waring, chairman and managing director of Joseph Lucas Ltd., and deputy chairman of the British Productivity Council.

Eleven papers are to be presented at the conference by leading specialists from Britain, America, Germany, Holland, France, Belgium and Italy.

Countries sending delegates include Germany, Holland, Belgium, Canada, Denmark, Czechoslovakia, Finland, India, Spain, Sweden, Switzerland and the U.S. Inquiries have been received also from a number of other countries including Russia and Japan.

Courses

A vacation course on 'The transformation of austenite,' will be held at the Metallurgy Department of the Battersea College of Technology on September 27-30. Fee £12 12s. (inclusive of luncheon, morning and afternoon refreshment.) Enrolment forms may be obtained from the Secretary (Metallurgy Courses), Battersea College of Technology, London, S.W.11.

The following evening courses are announced by the Borough Polytechnic, Borough Road, London, S.E.1, Division of Metal Science.

Refractories, their manufacture, properties and uses. Beginning on Friday, October 7, from 7 to 9 p.m. 10 meetings. Lecturer, Mr. L. R. Barrett, M.A., B.Sc., M.S., A.R.I.C., M.INST.F., M.INST.CERAM.

Metallurgy of semi-conductors. Beginning on Tuesday, October 25, from 7 to 9 p.m. 8 meetings. Lecturer, Mr. A. S. Abrahams M.A. (CANTAB.), A.I.M.

United Steel's university scholarship scheme

The United Steel Companies Ltd. are to offer up to ten university scholarships each year, tenable at any university in the United Kingdom. Each scholarship will be worth £500 per annum and its value will not be affected by parental income.

The scholarships will be offered to boys proceeding to a university to take full-time courses leading to approved degrees in appropriate branches of science or engineering. The company is also prepared to consider a limited number of applications from students of non-science subjects, recognizing that there is a place in industrial management for the good arts graduate.

Metal-fabricated nuclear components pass \$100 million sales mark

Sales of metal-fabricated nuclear reactor components and equipment are estimated to have reached \$101.9 million in 1958 according to statistics just released by the Atomic Industrial Forum, the association of the nuclear industry, in a special report entitled: *Business Statistics on the Atomic Industry, 1954-1958*. This estimate was projected from reported 1958 sales included in an AIF survey, which developed data on U.S. atomic industry sales of nuclear reactors, components, materials and services to government, industry and overseas markets for the five years from 1954 through 1958. The total sales estimated for the period for all the foregoing categories amounted to approximately \$1.5 billion, projected from a \$933 million total reported by close to 200 firms responding to the poll.

NEW FILMS

Lead the enduring metal

INTENDED to interest non-technical audiences, a film has been designed to offer general information on lead. After briefly tracing its history and ancient usage, there is a description of the metal's occurrence in the world today and how it is mined, smelted and refined.

The main sequences demonstrate the versatile role played by lead in the modern world and deal with applications which include sheet and pipe for building, paints, chemical engineering, lead shot, grids and oxides for storage batteries, type metals for printing, solder, electric cable sheaths and lead glass.

Lead's enduring properties and its well-known ability to resist corrosion are demonstrated in connection with its use in several fields. The metal's power to protect against harmful radiation hazards is described with particular reference to hospital and atomic energy plant and equipment. A closing sequence suggests that further new uses for lead will evolve as scientific discovery progresses.

Copies of the film (16 mm., 28 min., sound, colour) are loaned free of charge on application being made to the Lead Development Association, 18 Adam Street, London, W.C.2.

Advances in mechanical handling

A new 16-mm. sound/colour film, 'Accent on Mechanical Handling,' had its first showing in London recently.

Based upon sequences taken at recent mechanical handling exhibitions, the film depicts one or more examples of each of the various classes of mechanical-handling equipment.

The film is available free on loan from 'Mechanical Handling,' Dorset House, Stamford Street, London, S.E.1.

'Stone into steel' wins premier award at Venice

The United Steel Companies Ltd. have gained the premier award at the Venice Film Festival with their film 'Stone into steel' out of a total entry of 240 films from all over the world. Featuring the works of Appleby-Frodingham Steel Company, this colour documentary has no spoken commentary but the dramatic effect is heightened by a specially-composed musical score. The Golden Mercury trophy was presented to the producer, Mr. A. Frank Bundy, of Wallace Productions, by the President of the Venice Chamber of Commerce on July 30.

Copies of 'Stone into steel' in 16 mm. and 35 mm. are available on free loan on application to the public relations department of United Steel.

Die lubricant film

Graphoidal Developments Ltd. was formed two years ago to develop a new process of producing colloidal graphite dispersions, with the view to considerably speeding up the rate of production of this material and to lowering the cost of the finished product.

This object having been achieved, it is now possible for them to produce the dispersion for forging within twenty-four hours. Particular attention has been given to the application of a material suitable for the forging industry.

The company is at present engaged in the production of an 8 mm. colour ciné film intended to show some of the advantages obtained by the use of colloidal graphite as a die lubricant.

PEOPLE

AT A MEETING of the Council of the Iron and Steel Institute held on July 13, 1960, it was agreed unanimously to propose **Sir Charles Goodeve, O.B.E., D.Sc., F.R.S.**, for nomination as president-elect of the Institute at the autumn general meeting in November. Sir Charles will succeed Mr. W. F. Cartwright as president at the annual general meeting in May, 1961, and will thus be in office at the time of the Institute's proposed special meeting in the U.S.A. in October, 1961.

Charles Frederick Goodeve was born in Canada in 1904. He graduated at Manitoba University in 1919, where he carried out research on electrochemistry. In 1927 he was awarded a scholarship to University College, London, where he was appointed lecturer in physical chemistry in 1930 and reader in chemistry in 1937.

During World War II he served in the Royal Navy. He played an important part in countering the magnetic mine, and later developed and produced the 'Hedgehog' anti-submarine weapon. From 1942, as a deputy controller, he was in effective control of the whole research and development programme of the Royal Navy, and it was for his services in this connection that he was knighted in January, 1946.

Sir Charles left the Admiralty to become director of BISRA on its foundation in 1945. He has succeeded in combining his administrative duties with original research, and has made significant contributions to knowledge of the physical chemistry of metallurgical processes. He was president of the Faraday Society from 1950 to 1952 and president of the chemistry section of the British Association in 1956. In 1958-59 he was Master of the Worshipful Company of Salters. Sir Charles, who was elected a Fellow of the Royal Society in 1940, received the honorary degrees of Doctor of Science from the University of Manitoba in 1946 and from the University of Sheffield in 1956.

The Aluminium Development Association announces that **General Sir Geoffrey Bourne, K.C.B., K.B.E., C.M.G.**, has been appointed director-general to succeed Air Commodore W. Helmore, C.B.E., who recently retired after 14 years as director-general of the Association.

General Bourne has been A.D.C.-General to the Queen since 1959. He was educated at Rugby and the Royal Military Academy, Woolwich, and was commissioned into the Royal Artillery in 1923. After a distinguished war record he became G.O.C. Berlin (British Sector) 1949-51 and was Commandant of the Imperial Defence College 1958-59.

Dr. J. C. Hudson, D.Sc., D.I.C., A.R.C.S., F.I.M., who has been in charge of the British Iron and Steel Research Association's research on corrosion for the last 15 years—ever since the formation of the Association in 1945—will be retiring at the end of this month. He will, however, continue to have very close ties with the Association, since he has agreed to act as consultant to both the Corrosion Advice Bureau and the Chemistry Department.

Dr. Hudson was educated at Brighton College and at the Royal College of Science, to which he obtained a Royal scholarship in 1920. After taking his degree and associateship, he undertook research in physical chemistry. His first appointment was with the Atmospheric Corrosion Sub-Committee of the British Non-Ferrous Metals Research Association, where he worked in close association with Dr. W. H. J. Vernon, O.B.E. As a result of this work he was awarded the D.Sc. degree of the University of London. Then in 1929 he became in-

vestigator to the newly formed Corrosion Committee of the Iron and Steel Institute, since taken over by BISRA, and has continued in this post until now. He has also served as head of BISRA's Corrosion Advice Bureau since its formation in 1954.

Dr. Hudson has recently received two major awards in recognition of his contributions to corrosion engineering and ferrous metallurgy—the Frank Newman Speller Award for 1959 from the American National Association of Corrosion Engineers and the Sir Robert Hadfield Bronze Medal for 1960 from the Iron and Steel Institute.

The Council of the Institution of Production Engineers has approved the award for the year 1958-59 of the Silver Medal for the best paper presented to a section or region of the Institution by a member to **Mr. S. G. E. Nash, A.F.R.A.E.S., M.I.PRODE.**, head of materials laboratories, Bristol Aircraft Ltd., for his paper on 'Metallic materials and process development for aircraft applications.'

Mr. Nash commenced his engineering career in 1926 as an apprentice at the Royal Aircraft Establishment. In 1932 he joined the Bristol Aeroplane Co. as a test assistant, specializing later in mechanical testing and the application of materials. In 1944 he became the first head of the company's Aircraft Division structural research laboratory and, in 1950, quality manager. A return to development work followed in 1954 and in 1957 he became chief development engineer (metals). He took up his present position in 1958.

Since his election to membership of the Institution of Production Engineers in 1952 Mr. Nash has served more or less continuously on the Research and Technical Committee and is vice-chairman of its Material Utilization Committee. He also represents the Institution on several B.S.I. and other committees.

Mr. Richard Miles retired as chairman and managing director of Head Wrightson in January and has recently been given a presentation to mark his retirement by the board, staff and work people at the head office in Thorneby.

Mr. Miles joined the company in 1932 and was chairman and managing director for 10 years. He has been an active member of a number of national committees, including the Institute of British Foundrymen, Federation of British Industries, British Steel Founders' Association, British Engineers' Association, British Welding Research Association and many others, and was a director of the Nuclear Power Plant Co., National Research Development Corporation, F.C. Construction Co. and Franco Signs Ltd. and a number of Head Wrightson subsidiaries at home and abroad.

Head Wrightson & Co. Ltd. announces the following appointments among its subsidiary companies: **Mr. G. Jones** to be managing director of Head Wrightson Stampings Ltd., **Mr. W. H. Mather** as managing director and **Mr. G. Gowthorpe** as commercial director of the Head Wrightson Machine Co. Ltd.

Paul Granby & Co. Ltd. announces the appointment of **Mr. S. C. Winfield-Smith, D.S.O.**, as representative in the Midland area for the sale of all types of hot-forging equipment manufactured by Hasenclever, Lasco and Puddinghaus, sheet-metal working machinery made by Lasco, Wilhelmsburger and Verson, cold-rolling mills manufactured by Schmitz, and all presses and cold-

extrusion equipment made by Verson Allsteel Press Co., Chicago, U.S.A.

Mr. H. Roy Thielé has been appointed as deputy to the advertising manager of the Morgan Crucible Co. Ltd. Mr. Thielé was for many years personal assistant to the advertising manager of the Champion Sparking Plug Co. Ltd.

Mr. T. C. Bailey, sales manager, Industrial Fuels, Shell-Mex and B.P. Ltd., has retired and been succeeded by **Mr. E. Rendall**, divisional manager, North-Eastern Division, Leeds. **Mr. A. G. Simon**, sales manager, Irish Shell Ltd., is appointed divisional manager, North-Eastern Division.

Mr. T. C. Bailey joined the Anglo-Mexican Petroleum Co. Ltd. in 1915 and, after service in the Army, on the establishment of Shell-Mex Ltd. in 1921 he held appointments in Liverpool, Birmingham, Newcastle, Manchester and London. During the 1939-45 war Mr. Bailey served with the Petroleum Board, becoming principal technical adviser to fuel oil management. For the last six years he has been sales manager, Industrial Fuels, Shell-Mex and B.P. Ltd.

Mr. Bailey has been closely associated with the development of oil firing for open-hearth furnaces in the steel industry since the war. President of the Institute of Fuel in 1958-59, he has been the author of a number of papers on the use of oil fuels.

Mr. E. Rendall joined Shell-Mex Ltd. in 1927 and held appointments in London and Hull. After service as a technical officer in the R.A.F. from 1940 to 1945, Mr. Rendall held an appointment with the Petroleum Board. His subsequent career with Shell-Mex and B.P. Ltd. has included periods as Birmingham branch manager, sales manager, Scottish Oils and Shell-Mex Ltd., and divisional manager, North-Eastern Division, Shell-Mex and B.P. Ltd.

Mr. W. E. A. Redfearn has resigned as chairman of the Alloy Steels Association and is succeeded by Mr. R. Bavister.

Mr. Redfearn is a director of English Steel Corporation Ltd., English Steel Rolling Mills Corporation Ltd., and managing director of English Steel Forge and Engineering Corporation Ltd., and has been chairman of the Alloy Steels Association for ten years. He is a past president of the NADFS.

Mr. Bavister is a director and commercial manager of Samuel Fox & Co. Ltd.

At a recent meeting of the Council of the British Welding Research Association, **Mr. J. D. D. Morgan** and **Dr. L. M. Wyatt** were appointed to the Research Board. Mr. Morgan is with the General Chemical Division of Imperial Chemical Industries Ltd., and is the chairman of the I.C.I. Welding Panel. Dr. Wyatt is the chief metallurgist of the Central Electricity Generating Board.

Mr. Henry Snow has been appointed industrial liaison officer of the Coil Spring Federation Research Organization. His job will be to improve the lines of communication between the laboratory and the factories. He will be 'selling' research not only to the comparatively tiny spring-making industry itself, which employs only about 7,000 people, but to all the many user industries who are also members of the organization.

Edgar Allen & Co. Ltd. announces the appointment of **Mr. W. H. Everard**, deputy general manager of the Foundry Division of the company, as president of the British Electric Steel Makers' Guild.

The Guild, which was formed in 1957, aims to promote

progress in the manufacture of steel for commercial purposes in electric melting furnaces and to encourage exchange of information between members.

G.W.B. FURNACES LTD. announces that following the untimely death of **Mr. A. V. Francis**, Furnace Division manager, the following appointments have been made:

The new manager of the Furnace Division will be **Mr. W. L. Harrison**, who has spent many years in the furnace industry, particularly on the melting side. In future the division will operate as two units, one dealing with melting and one with heat-treatment processes. **Mr. J. Simpson** will be sales manager (melting) and **Mr. J. Nicholls** will be sales manager (heat treatment). Both Mr. Simpson and Mr. Nicholls have been with the company for many years, the former for some time being manager of the Canadian branch.

Finally, **Mr. A. Head**, for many years chief draughtsman, has been appointed chief designer.

Smith's Stamping Works (Coventry) Ltd. announces that following the death of Mr. S. Bramhall, M.B.E., chairman and managing director, **Mr. H. B. T. Wilde** has been appointed chairman and **Mr. H. F. W. Perry** and **Mr. H. M. H. Fox** joint managing directors.

Mr. W. N. Smith, who has been with the company for over 25 years, has been elected a director. **Mr. G. Warriner**, after over 30 years' service at the Lincoln works, has been appointed a director of Smith-Clayton Forge Ltd.

Mr. A. W. Wallbank, B.Sc., F.R.I.C., managing director of Ionic Plating Co. Ltd., Grove Street, Birmingham, 18, the Metal Finishing Division of the G.K.N. Group of Companies, received the Gold Medal award at the American Electroplaters' Society's Convention at Los Angeles last month. The prize is the Carl E. Heussner A.E.S. Gold Medal Award (the Society's Highest paper award) for his paper, 'Barrel plating with special consideration to protection of thread diameters.'

OBITUARY

We deeply regret having to report the death of **Mr. Sidney Bramhall**, M.B.E., chairman and managing director of Smith's Stamping Works (Coventry) Ltd., at the age of 68.

Mr. Bramhall was born in Sheffield and was previously associated with William Jessop & Sons Ltd., Cammell Laird & Co. Ltd. and English Steel Corporation, of Sheffield. He joined a subsidiary of Smith's Stamping Works (Smith's Clayton Forge Ltd., of Lincoln) in 1934 and became a director of both these firms in 1936. In May, 1954, he became chairman of Smith's Stamping Works (Coventry) Ltd. He was president of the National Association of Drop Forgers and Stampers for two years (1954 and 1955) and has always been an enthusiastic supporter of the Association and regular attendant at its functions in spite of recent ill health.

Mr. Bramhall received his M.B.E. for wartime services to industry in general.

We offer our sincere sympathy to his widow and three daughters.

Mr. W. M. Grainger, director and secretary of Walter Somers Ltd., died recently.

Mr. Grainger joined the company in 1946 as assistant secretary on his return from H.M. Forces. Several years later he was made secretary and in 1959 was appointed to the board of directors. Mr. Grainger was also a director of the subsidiary company, Walter Somers (Materials Handling) Ltd.

INSTRUMENTATION

Constant-voltage source for Kent instruments

The type 3 reference unit is a solid-state d.c. reference source designed, developed and produced by George Kent Ltd., of Luton, Beds., England, for use with the company's range of electronic self-balancing recorders, indicators and controllers. A compact plug-in assembly operating directly from the normal a.c. mains supply, it provides an extremely stable, ripple-free output of 5.0 mA. at 5 volts for the slidewire of the instrument measuring circuit.

Two Zener-diode stages in cascade reduce the effects of normal supply variations by a factor of at least 200, while individual temperature compensation of each of these stages ensures that the output is substantially independent of changes in ambient temperature between 10 and 70°C. An inherent advantage is that the unit continues to operate with only slightly reduced accuracy if the supply voltage is drastically reduced, even to as little as 30% of its nominal value.

Beryllium monitor for industrial health

A new beryllium monitor has been designed and developed by M. S. W. Webb, R. J. Webb and P. C. Wild of the United Kingdom Atomic Energy Authority's Woolwich out-station and is being manufactured and marketed by Winston Electronics Ltd., Shepperton, Middlesex. In conjunction with Dr. A. H. C. P. Gillieson of the Atomic Energy Establishment, Harwell, Winston Electronics produced the first, semi-automatic instrument six years ago, and the new instrument is more completely automatic and rapid in operation.

It is essential to the health of persons employed in workshops and laboratories using beryllium, its ores and alloys, that the beryllium concentration should not exceed two millionths g.m.⁻³. It is essential also that sudden, or unexpected increases in beryllium concentration should be quickly indicated, as with this new instrument, and that some kind of warning should be given automatically.

The toxic threshold for beryllium is one-seventy-fifth that of lead and one-fiftieth of the next most toxic dusts, mercury and selenium. For an 8-h. shift the beryllium toxic threshold is 2 $\mu\text{g. m.}^{-3}$ and at no time should it momentarily rise to more than 25 $\mu\text{g. m.}^{-3}$.

The '4W' beryllium monitor for the detection of beryllium aerosols visually records on a graph every 60 sec. the beryllium content of a fresh sample of the atmosphere in which the instrument is present.

It gives quantitative results over the range of 1 to 75 millionths g.m.⁻³, irrespective of the particle size or chemical composition of the beryllium atmospheric dust.

The monitor is a fully quantitative instrument for the optical estimation of the element. Its accuracy is now $\pm 15\%$ for beryllium metal and $\pm 30\%$ for beryllium compounds and alloys.

The sampling head is portable and connected by an air hose, which also contains the connecting leads, to the estimating console. This improvement enables the atmosphere to be sampled at an operator's inhalation level when working the metal or its alloys.

The air to be sampled is drawn through a chamber in which any beryllium present is optically excited as it passes through a triggered alternating current arc. The chamber has been carefully designed aerodynamically to eliminate pockets of beryllium concentration.

The ultra-violet radiations from the excited beryllium are then resolved into their components by means of a spectograph with a high dispersive power.

The intensity of the beryllium doublet at 3,130 Å, which is proportional to the concentration of beryllium present in the air sample, is measured photo-electrically and then the ratio of the intensity of this doublet to the intensity of the adjacent background is recorded on a chart, i.e. beryllium line background.

The automatic calibration is achieved by the use of a simple condensed spark discharge between a beryllium-copper electrode and a copper counter electrode producing an aerosol containing a uniform concentration of beryllium at a constant rate, the air for this being drawn through special filters. The monitor is fully automatic and it operates on a predetermined cycle in which it is first calibrated and then records a series of results at 1-min. intervals.

Rapid-response direct-reading portable thermometer

The MAVOTHERM is a comparatively new portable instrument for the measurement of temperatures in solids, liquids or gases. Ideally suitable for use in both laboratories and workshops for rapid accurate measurement of ambient or static temperatures, the MAVOTHERM combines accuracy and speed with safety and convenience. Reading time is between 3 and 4 sec. and accurate to ± 2 °C.

The instrument is battery operated and, under normal load conditions, its 1.5-volt battery will last for nearly a year. It has two ranges (-20°C. to +90°C. and +90°C. to +200°C.) and the scale readings are calibrated in degrees centigrade. Two calibrated probes specially designed for easy access to difficult locations are supplied,

1 The Mavotherm portable thermometer

and the whole instrument is contained within a strongly made plastic case. The MAVOTHERM is manufactured by P. Gossen of Germany and distributed in the United Kingdom by Aveye Electric Ltd.

15-MeV. mobile betatron

For non-destructive testing of steel thicknesses of up to 4 in., the majority of supervoltage X-ray units and radioactive isotopes currently used are sufficient in power. Above this thickness, however, exposure times become unacceptably long. At the same time, as thicker fabrications in steel become more common and as radiography is extended to the testing of other types of material such as reinforced concrete, the need arises for a powerful X-ray generator which possesses an extremely high degree of sensitivity when dealing with thick structures.

The Siemens 15-MeV. mobile betatron, which has just been introduced to the market by Pantak Ltd., of Vale Road, Windsor, Berks., comprises essentially an electromagnet shaped in the form of a shell-type transformer which contains an air gap in its middle leg. The function of this a.c. excited transformer is to create a magnetic flux in which electrons are accelerated almost



2 The Siemens 15-MeV. mobile betatron

to the speed of light in a doughnut-shaped accelerator tube which surrounds the electromagnet. The flux produced by the field varies rapidly in shape from the injection stage, when electrons are injected from a cathode filament, through four following stages of deflection and transfer into the magnetic field, a packing phase, a distribution phase, a phase when the flux is constricted, this leading finally to the emission of electrons which strike a platinum target and thus give rise to very high-energy X-rays.

The magnet which carries out this process has two rotation-symmetrical poles with parallel faces arranged as a two-legged yoke, the distribution of the field produced being governed by pole piles which are clamped between the poles. These pole pieces are critically important in that they govern the location of the orbit of the flux and the strength of the magnetic force which stabilizes it. Both the pieces and the surrounding accelerator tube are pneumatically pressed into contact with the magnetic yoke which gives stiffness and protection during transport in addition to damping down both transformer noise and vibration.

The accelerator tube is constructed of a ceramic known as frequenta, the inner wall of this doughnut tube being coated with a metal film. The materials used are degassed and the tube is evacuated to the low pressure of 10^{-6} torr. The tube is sealed by a 30-micra-thick nickel foil, through which electrons accelerated in the flux by the magnet may leave if the production of free electrons is

required. For X-ray work, however, these electrons are arranged to graze a platinum target which is fixed to the inner wall of the tube, the resultant X-ray beam being taken out through the frequenta wall of the tube.

The magnet coils are energized by a regulating transformer connected directly to a 50-cycle 3-phase mains supply, the consumption amounting to approximately 12 kVA, and a regulator is employed to correct mains voltage fluctuations between 180 and 240 volts. The betatron is operated from a small portable control case, and with a total weight of under 3½ tons it may be readily transported by truck from site to site.

The substantial savings in exposure time which can be made with this new tool are shown for example by the fact that exposures for steel thickness of between 6-8 in. are only a few minutes, whilst that for a 16-in. thickness of steel is between 1 and 2 h., as compared with an exposure of several days which would be required even with a supervoltage X-ray tube of the conventional type. Apart from this saving, a new technique of radiographing a workpiece whilst still hot is opened up and, since the cooling-for-examination stage is one where flaws may develop, the elimination of this stage in thick section fabrication will lead to the prevention of costly flaws in this type of work as further fabrication can follow inspection without the need for a fluctuating temperature gradient. The third point of note is the fact that a magnification technique, giving an image of up to three times the size of the flaw, can be used.

New high-range Pressuretrol

A recent addition to the Honeywell range of pressure controllers is the new industrial Pressuretrol, model P428, designed for general applications requiring control or limiting of gas or liquid pressures up to 3,000 lb./sq.in. It can operate alone as a controller or with other instruments for limit, alarm or signalling service. Five ranges are available, from 30-300 to 300-3,000 lb./sq. in.

The industrial Pressuretrol consists of a mercury switch operated by a corrosion-resistant Bourdon tube, enclosed in a heavy steel case which resists dust, weather and corrosion. Pressure-setting knobs are conveniently located on the front; tamper-proof knobs can be provided.

Switch action is SPST, make or break on pressure rise, depending on the model. The position of the mercury switch is visible through a window in the front of the case. An explosion-proof model is available for use in hazardous atmospheres. Full details are given on specification S1011-3 available on request from Honeywell Controls Ltd., Ruislip Road East, Greenford, Middlesex (WAXlow 2333).



3 Honeywell
high-range
Pressuretrol

NEW PLANT

Ultrasonic cleaning for degreasing plants

I.C.I. is now marketing a series of ultrasonic cleaning plants designed for use with trichloroethylene. Ultrasonic generating equipment, made by Dawe Instruments Ltd., 99 Uxbridge Road, London, W.5, is connected to a sealed stainless-steel transducer unit containing barium titanate elements submerged in cool trichloroethylene in one compartment of the plant. Here, electrical impulses at a frequency of 40 kc/s. are changed to mechanical vibrations which produce 'cavitation' in the solvent. Cavitation means the alternate formation and violent collapse, at a very high rate, of minute cavities in the liquid, giving an intense scouring action at the surfaces of the work. The high solvent power of trichloroethylene on oil and grease contamination is assisted by the mechanical effect of the scouring, so that insoluble solid particles are effectively detached, even from cracks and pits on metal surfaces. The solvent in the ultrasonic cleaning compartment is circulated by a pump through a filter, where solids are collected, and back to the same compartment. The filter is easily accessible for renewal.

The other compartment in the plant holds electrically heated boiling trichloroethylene below a vapour layer. Vapour rises to the condensing coils, falls as liquid into the ultrasonic compartment and returns to the boiling liquor compartment by over-flowing the weir plate.

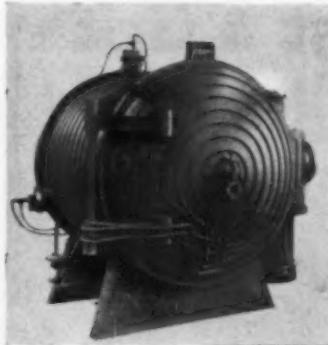
The route of articles through the plant varies according to their condition. Most of the work for which the ULV1 is appropriate is not heavily greased or oiled and can go straight into the ultrasonic cleaning compartment, and then into the vapour above the boiling liquor. Grossly contaminated work should be given a preliminary dip in boiling liquor; in the larger plants three compartments are provided so that a steady flow of work can be maintained. The final immersion in vapour is desirable for safety and economy, as it ensures that work is dry when it leaves the plant.

Safety features include rim ventilation equipment and a thermostat which cuts off the heat if the water fails or if the solvent level falls dangerously low.

Automatic high-vacuum heat-treating furnace

An automatic, high-vacuum, cold-wall-type furnace with a work space 26 in. wide, 26 in. deep and 24 in. high, has recently been developed by Ipsen Industries Inc., Rockford, Illinois, U.S.A. The general-purpose vacuum heat-treating unit will be used for high-temperature copper brazing and heat treating of gas turbine parts and assemblies. Like other standard Ipsen vacuum heat-treating units, the furnace can be used for hardening, tempering, drawing and annealing refractory metals, stainless and special alloy steels.

The furnace, series V-5-750, has an operating temperature of 2,600°F., a maximum operating pressure of 0.1 micron Hg, and takes a power supply of 150 kW/h. It has a transformer built in for operating 110-V. control circuits. Total floor space required is 10 ft. 10 in. × 13 ft. 10½ in., which includes the furnace, the vacuum system, control panel and power panel. The furnace is charged horizontally and has a molybdenum hearth. The charging door is side hinged and is equipped with a sight glass for viewing the work during heating. The pumping system consists of a 120-cu.ft./min. mechanical pump and a 16-in.-dia. vapour diffusion pump capable of a pump-down time from atmosphere to 1 micron Hg in less than 20 min. The furnace employs graphite heating elements (molybdenum heating elements are optional) and multiple radiation shields which are easily removed for maintenance and replacement purposes.



1 Ipsen
automatic high-
vacuum furnace

The power panel of the V-5-750 vacuum furnace houses the main power transformers, the main contactor, the current transformer for amperes and voltage indicators. The control panel has a switch allowing the furnace to be set for manual or automatic operation at the will of the operator. The entire heat-treating sequence, including time at temperature and cooling cycle, can be operated automatically. All operations or any single operation can be changed from automatic to manual at any time during the sequence.

The cooling system consists of a vacuum-sealed fan mounted within the furnace to provide forced circulation of an inert gas over the work during the cooling cycle. Cooling from 2,600°F. to less than 400°F. is accomplished in less than 1½ h.

Safety features include a thermocouple vacuum gauge controller which serves as a safety switch to set off an alarm if the vacuum system does not reach 100 microns Hg at the end of a pre-set time. It is interlocked with the heating circuit to prevent power from being applied to the furnace element if vacuum is above 100 microns Hg.

Ipsen Inc. is represented in the U.K. by Mr. T. W. Ruffle, 53 Victoria Road, Surbiton, Surrey.

Machinable cemented carbides

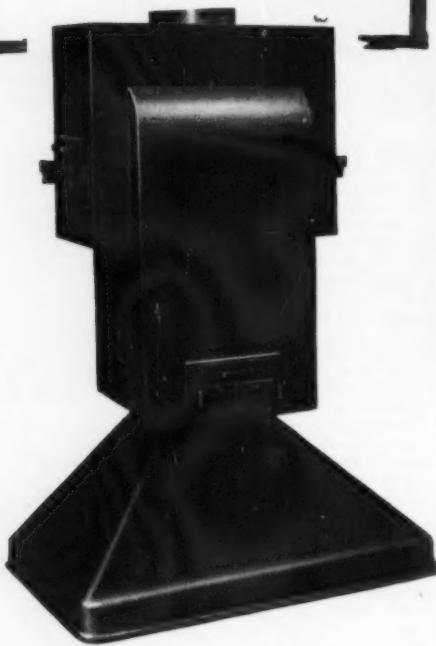
Tools which have to be hard to withstand wear, parts such as valves and valve seats, and knives and nozzles for the chemical and food processing industries, which have to resist corrosion or high temperatures, are frequently produced in tungsten carbide or cobalt-base alloys. The production of such components on a 'one-off' basis is not easy since it is normally necessary to form them by casting or by diamond grinding.

Developments of powder metallurgy techniques in the U.S.A. have led to the development by Chromalloy Corporation, Yonkers, N.Y., U.S.A. of a new form of cemented carbide which overcomes these difficulties. The material consists of crystals of extremely hard titanium carbide embedded in a matrix of steel, which in one variety is a stainless steel and in another is a low-alloy steel. In the annealed state these materials may be worked by conventional techniques of machining, welding and grinding. Components may therefore be produced as desired in the machine shop from stock blanks. Upon completion, the material is easily heat treated by oil quenching to develop the degree of hardness required.

The material, Ferro-Tic S, is available in the U.K. from John Rigby & Sons Ltd., Cleckheaton, Yorkshire.

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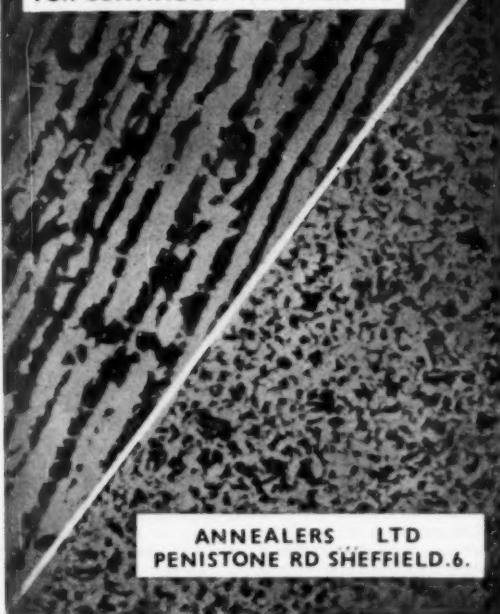
This photograph shows an Alldays & Onions UF/I Dust Collector, primarily for use with hearths and designed to meet the requirements of the Clean Air Act.

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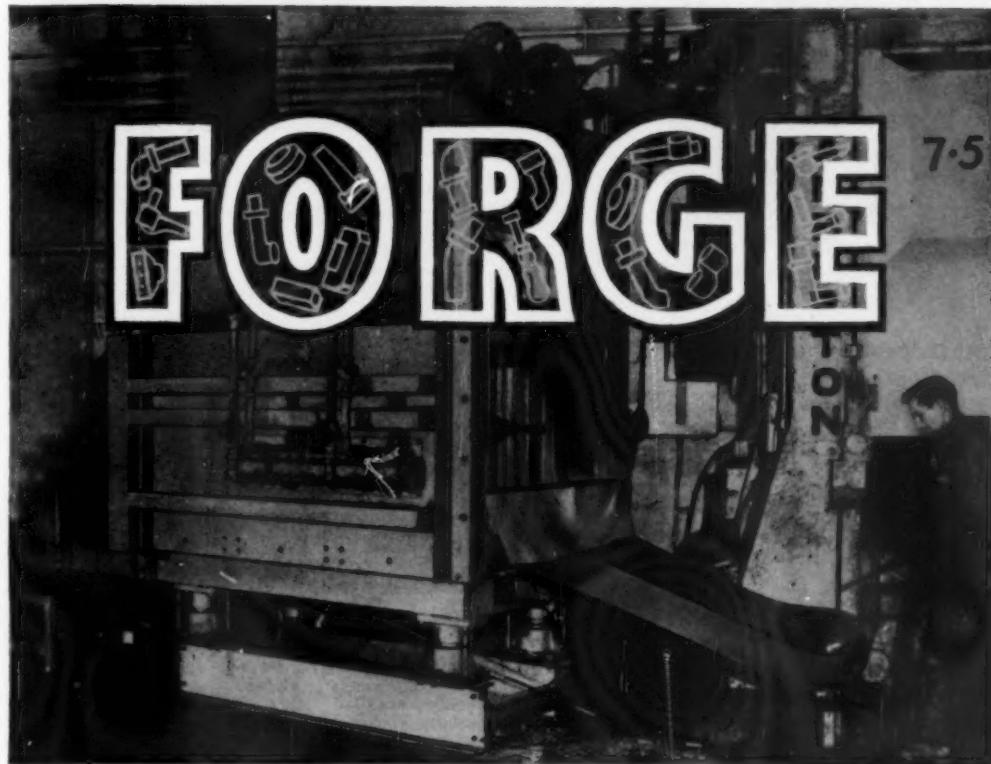
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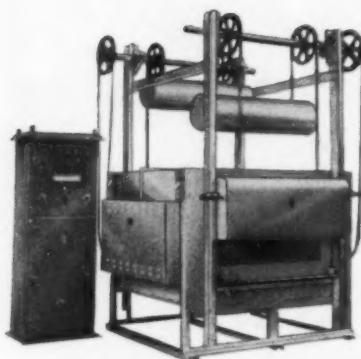
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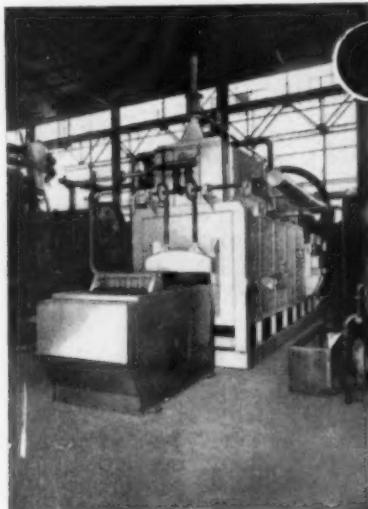
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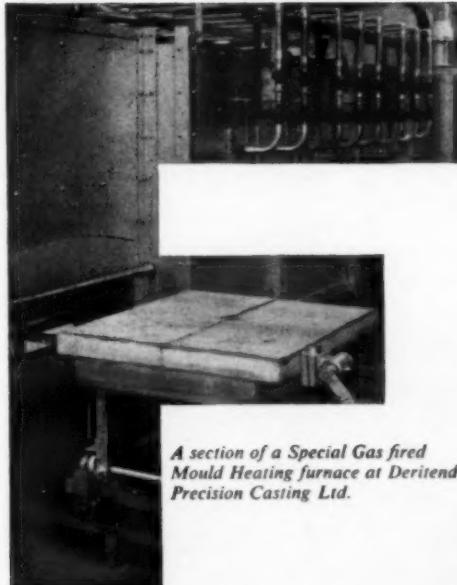
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INDEX TO ADVERTISERS

Abbey Heat Treatments Ltd.	34	Ether Ltd.	9	Massey, B. & S. Ltd.	2
A.C.E.C.	10	Eumuco (England) Ltd.	—	Mellows & Co. Ltd.	—
Acheson Colloids Ltd.	—	Fel Electric Ltd.	—	Metalectric Furnaces Ltd.	16
A.E.I.—Birlec Ltd.	26	Firth, Thos., & John Brown Ltd.	14	Modern Furnaces & Stoves Ltd.	—
Alldays & Onions Ltd.	30	Firth-Derihon Stampings Ltd.	7	Mond Nickel Co. Ltd.	—
Andrews, Thos., & Co. Ltd.	34	Flame Hardeners Ltd.	35	Morgan Refractories Ltd.	—
Annealers Ltd.	30	Folias Graphite Products Ltd.	—	Morris, B. O., Ltd.	—
Barlow-Whitney Ltd.	32	Franklin Furnace Co. Ltd.	35	Newall, A. F., & Co. Ltd.	—
Birlec-Efco (Melting) Ltd.	—	Fuel Furnaces Ltd.	—	Nitralloy Ltd.	—
Brayshaw Furnaces Ltd.	—	Gas Council	38	Nu-Way Heating Plants Ltd.	—
Briggs Bros. (Engineers) Ltd.	30	General Electric Co. Ltd.	19	Priest Furnaces Ltd.	20
British Furnaces Ltd.	11	General Refractories Ltd.	—	Shell-Mex & B.P. Gates Ltd.	—
British Resistor Co. Ltd., The	—	Gibson Brothers Ltd.	32	Siemens-Schuckert (G.B.) Ltd.	—
Broadbent, Thos., & Sons Ltd.	33	Gibbons (Dudley) Ltd.	—	Siemag Electrical Instrument Co. Ltd.	—
Burbridge, H., & Son Ltd.	—	Granby, Paul, & Co. Ltd.	5	Smethwick Drop Forgings Ltd.	—
Burdon Furnaces Ltd.	—	Graphtal Developments Ltd.	35	Somers, Walter, Ltd.	—
Calorizing Corporation of Great	—	G.W.B. Furnaces Ltd.	4	Special Steel Co. Ltd.	—
Britain Ltd. (The)	—	Hedin Ltd.	34	Storck Engineering Ltd.	6
Cambridge Instrument Co. Ltd.	—	Herbert, A., Ltd.	21	Thermal Equipment & Engineering	31
Coventry Machine Tool Works Ltd.	—	Honeywell Controls Ltd.	—	Co. Ltd.	—
Cronite Foundry Co. Ltd. (The)	37	Hydraulic Engineering Co. Ltd.	—	Thompson, John, (Dudley) Ltd.	—
Delapena & Son Ltd.	—	I.C.I. Ltd.	—	Thompson, Joseph, (Sheffield) Ltd.	25
Dohm Ltd.	—	Incandescent Heat Co. Ltd. (The)	24	Thompson L'Hospied & Co. Ltd.	1
Doncaster, Daniel, & Sons Ltd.	—	Integra, Leeds & Northrup Ltd.	15	United Steel Companies Ltd.	—
Dowding & Doll Ltd.	18	Johnson Foster, H., Ltd.	36	Vacuum Industrial Applications Ltd.	—
Dowson & Mason Ltd.	—	Kelvin & Hughes (Industrial) Ltd.	—	Vaughan, Edgar, & Co. Ltd.	—
Eco-Edwards Vacuum Metallurgy	—	Kiveton Park Steel & Wire Works	—	Wickman Ltd.	—
Ltd.	—	Ltd.	—	Wiggin, Henry, & Co. Ltd.	7
Electric Furnace Co. Ltd.	—	Lafarge Aluminous Cement Co. Ltd.	12	Wild-Barfield Electric Furnaces Ltd.	3, 13
Electric Resistance Furnace Co. Ltd.	8	Langley Forge Co. Ltd.	23	Wilkins & Mitchell Ltd.	22
Electrical Development Assn.	—	Manchester Furnaces Ltd.	—	Workington Iron & Steel Co.	—
Electro Heat Treatments Ltd.	—	—	—	—	—
English Electric Co. Ltd.	—	—	—	—	—
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E.N.V. Engineering Co. Ltd.	28	—	—	—	—

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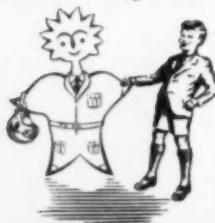
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